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UPGRADING OF SYNTHETIC CRUDE DISTILLATES
WITH COMMERCIAL HYDROTREATING CATALYSTS

Final Report

by

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Project Manager
Stearns Catalytic Ltd.
Calgary, Alberta, Canada



DSS File No. 06SQ. 23440-9318
Contract Serial No. 0SQ85-00031

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Work on the project was conducted under the auspices of the Canada Centre
for Mineral and Energy Technology, Energy, Mines and Resources Canada.

DSS File No. 06SQ.23440-4-9318
DSS Contract Serial No. 0SQ85-00031
Stearns Catalytic Project: 4046
CANMET Scientific Authority: Dr. M.V.C. Sekhar

February, 1987

MICROMEDIA

158 pp

4046/02/87

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EXECUTIVE SUMMARY

This contract, Upgrading of Synthetic Crude Distillates with Commercial Hydrotreating Catalysts, was carried out under auspices of Energy Research Laboratory of CANMET by Stearns Catalytic Ltd. and Saskatchewan Research Council as the subcontractor for the experimental work. This report details findings, results, conclusions and recommendations.

The backgrounds and objective that primed this significant technology development project is presented in the beginning of Chapter I. Major tasks involved to achieve the objective were outlined as well. Chapter II, SRC's Subcontractor Report, gives a comprehensive account of experimental work, including results and conclusions of the hydrotreating tests.

Presentation of a design of a down-sized Canadian Hydrotreating Test Centre (CHTC) is done in Chapter III. It includes all the essential scope required for future detailed implementation of such a facility plant.

Completing the project report is the Conclusions and Recommendations compiled in Chapter IV which can be summarized as follows:

- Hydrotreatability of Canadian Synthetic Crude Distillates with conventional, commercial catalysts has been proven,
- Canadian hydrotreating expertise has, as well, been established and enhanced by this contract work,

Ce contrat, pour l'amélioration du distillat synthétique brut par l'hydrotraitement de catalyses commerciaux s'est produit sous les auspices du Laboratoire des Recherches d'Énergie de CANMET, par Stearns Catalytic Ltée. et le Conseil de Recherches de Saskatchewan comme sous-entrepreneur au stade expérimental. Ce rapport donne les résultats, conclusions et estimations recommandables de cette expérience.

Chapitre I nous décrit le fondement et les objectifs du développement primordial de cette technologie importante, ainsi que les tâches essentielles pour atteindre ces mêmes objectifs. Chapitre II, le rapport du sous-entrepreneur (le Conseil de Recherches de Saskatchewan) est un compte-rendu compréhensible de cette expérience, ainsi que les résultats et conclusions des examens d'hydrotraitement.

Une présentation des plans pour le développement d'un "mini" Centre Canadien pour Examens d'Hydrotraitement est donnée dans Chapitre III. Ce chapitre nous donne aussi un aperçu des moyens requis pour la réalisation d'un projet d'une telle envergure.

Complétant le rapport de ce projet sont les conclusions et estimations recommandables compilées dans le chapitre IV, qui peuvent être résumer comme suit:

- L'abilité de l'hydrotraitement de distillats synthétiques bruts commerciaux Canadiens avec les catalyses et conventionnels a été prouvé;
- L'expertise canadienne en l'hydrotraitement a été établie et s'est accrue grâce à ce contrat;

EXECUTIVE SUMMARY (cont'd)

- Areas of improvement for continuing technology development are identified and recommended and,
- Pour un développement technologique continu, les domaines ayant besoin d'amélioration ont été identifiés et recommandés, et;
- A CHTC as recommended for immediate implementation would combine the development needs for R & D and serve industrial users.
- Un "Centre Canadien pour Examens d'Hydrotraitement" tel que recommandé pour implantation immédiate devrait réunir les besoins de développement pour Recherches et Développement, ainsi que servir les consommateurs industriels.

Design drawings, data and technical files are attached in the Appendices.

Les croquis, documents et dossiers techniques sont joints dans l'Appendice.

I. INTRODUCTION

I. INTRODUCTION

The Project at the beginning phase was conceived to be the foundation on which a genuine Canadian Hydrotreating Technology is to be further developed and built up. This was fully justifiable at a time of technical needs and, as well, economic incentives. While the short-term economic drive may have been slackened due to oil price drop, the need for further technology advancement to a higher stage remains as valid as ever. And indeed the statement:

"As the supply of high quality natural crude oils decline; the increased use of synthetic crudes will become more of a necessity. Research in the area of utilization of synthetic crudes is of special and continuous interest -----" for Appendix "A", REQUEST PROPOSAL by DSS/CANMET

has concisely summed it up - concurring with our opinion then and TODAY.

In spite of the change of economic climate, the work under this Contract has been carried out to its possible fullest intent, encompassing tasks of Research, Technology Evaluation and Engineering Design based on the former two tasks. CANMET/EMR is now in a good position to extend an effort towards achieving the longer term objective set forth in the original RFP.

The work under the direction of Dr. M.V.C. Sekhar, the Scientific Authority was a joint product of Stearns Catalytic Ltd. and Petroleum Division of Saskatchewan Research Council. SRC, the Subcontractor, performed all the hydrotreating tests and was responsible for the analytic work as well. Personnel who had devoted or contributed to the project are listed in Appendix G.

Various key tasks are presented separately in the following chapters, with the last Chapter 6 summarizing Conclusions and Recommendations.

By way of introduction and as a matter of record, three important subject topics will be presented in this chapter:

- | | |
|-----------------------|---|
| 1.0 OBJECTIVE: | What were set forth to be achieved. |
| 2.0 SCOPE OF WORK: | What were to be done to achieve the Objective. |
| 3.0 OVERVIEW OF WORK: | What have been done as measured against the Objectives and Scope. |

I. INTRODUCTION (cont'd)

1. OBJECTIVES

As a significant initiative, the project has immediate, specific objectives and, more importantly, a longer term perspective.

The specific objectives as outlined in DSS/CANMET's RFP and adopted in our contract proposal are:

- (a) Testing of Conventional Hydrotreating Technology against synthetic crude or tarsand distillates and the like feedstocks from heavy oil-based derivatives.
- (b) Assessing the suitability for downstream processing of the hydrotreated product.

Operating and analytic database must be developed and established in order to achieve the above.

The immediate term goal is, in short, to evaluate applicability of conventionally used technology to hydrotreating the rather "unconventional" feedstocks.

The project was also perceived from a larger, longer term perspective standpoint, as an embryo for

- (c) Development of Canadian Hydrotreating Expertise, with a specific reference to Canadian crude feedstocks.
- (d) Conceptual Design or Feasibility Study of a distillate hydrotreating testing centre.

It should be noted that only the success of short-term objectives (a) and (b) can positively lead to achievement of the longer term objectives (c) and (d). However, we have believed from the proposal stage that based on our experience and the trust of SRC's expertise both the short and long term objectives will be achieved. As such, the Project Scope of Work was then prepared for it.

I. INTRODUCTION (cont'd)

2. SCOPE OF WORK

Along the established objectives in Section 1, the Scope has entailed performance, in sequence of execution, of following primary tasks.

- 1) Design, Procurement and Setup the Hydrotreating Test System. This includes acquisition of components and equipment required for the experimental facilities.
- 2) Selection and Acquisition of Synthetic Crude Distillate and Commercial Catalysts in consultation with and subject to approval of Scientific Authority of CANMET.
- 3) Analyses of the Selected Feedstock and Catalysts.
- 4) Performing Hydrotreating Screening Tests for sets of operating parameters (P, T and LHSV) and runs durations being agreed upon with CANMET.
- 5) Analyses of Gas and Liquid Products.
- 6) Comprehensive Data Analyses on which selection of the best performing catalyst and operating conditions are based for the next task.
- 7) Performing Detailed Hydrotreating tests.
- 8) Products Analyses as in 5), Data Analyses as in 6) and Catalyst Micro-Activity-Test (MAT).
- 9) Conceptual Design/Study for a Canadian Hydrotreating Test Centre (CHTC).

The decision to carry out Task 9) was made after completion of Task 6).

For ease of reference, catalysts properties are compiled in Appendix A.

I. INTRODUCTION (cont'd)

3. OVERVIEW OF WORK ACCOMPLISHED

Results and details of work done are presented in the next several chapters, with experimental work being presented separately in SRC's Subcontractor Report attached to Chapter II as part of this Final Report. Briefly described in this section is intended to give an overview only.

The experimental system was quickly set-up, with a Milton Roy (feed) pump and two load cells purchased and tested. The required supporting facilities, such as those for analyses and a distillation unit, were identified and properly maintained. Section II of the Subcontractor Report gives detailed account and review of the experimental systems. This completed Task 1).

At a project meeting with CANMET, Suncor Coker Gas Oil as the feedstock and five commercial hydrotreating catalysts were recommended to and accepted by the Scientific Authority. Acquisition and preliminary assessment of the catalysts and feedstock analysis were carried out, establishing the database for subsequent evaluation. Presulphiding of the first catalyst to be used in the Screening Test was also done.

The Hydrotreating Screening Tests, which consumed most of the project time, were performed for the following processing parameters

Pressure:	6 MPa, 10.5 MPa
Temperature:	360°C, 370°C, 380°C
LHSV:	0.5, 0.75, 1.0

at a fixed hydrogen feedrate of 900 c.c./c.c. liquid feed. A total of nineteen (19) runs for each catalyst were completed for Task 4). This was followed by gas and liquid product samples analyses (Task 5).

The Screening Tests data was collected, collated and correlated with processing conditions. Material balance for each run was performed. Catalyst performance evaluation based on

- desulphurization
- denitrogenation
- liquid product density
- hydrogen consumption

I. INTRODUCTION (cont'd)

3. OVERVIEW OF WORK ACCOMPLISHED (cont'd)

led to the selection of Shell S-424 being the overall best catalyst for Task 7), detailed Hydrotreating tests. Run conditions and duration for Task 7) were recommended to and approved by CANMET. Task 6) was therefore completed. At this point, it had become clear that the conventional hydrotreating technique can be applied, pending on improvement and scale-up, to Canadian Synthetic Crude Distillates. And the experience and expertise gained from this project would enable a valuable input to a conceptual design of a more comprehensive hydrotreating facility system. Task 9) was initiated.

Detailed Hydrotreating Tests (Task 7) and products analyses were carried in the same manner as screening tests, with variations in run conditions and presulphiding procedure. MAT tests using ASTM procedure was also carried out to complete Task 8).

The Design Study, Task 9), which was conceived during or soon after Task 6) was expanded to a more comprehensive engineering work. Completed work includes:

- Process Flow Diagram c/w Mass Balance based on experimental results (Appendix C).
- Piping and Instrumentation Diagram (Appendix D).
- Facility Plot Plan (Appendix E).
- Equipment List compiling essential process design data (Appendix F).

for a Canadian Hydrotreating Test Centre (CHTC).

In summary, all the tasks under the Scope of Work that are required to achieve the pre-established project objectives have been successfully executed. CANMET/EMR is now in a good position to implement the results to attain the viable long term objective.

II. HYDROTREATING TESTS

- SUBCONTRACTOR REPORT BY
SASKATCHEWAN RESEARCH COUNCIL

II. HYDROTREATING TESTS

The experimental work was subcontracted to Petroleum Division, Saskatchewan Research Council (SRC), Regina, Saskatchewan. The work was consisted of, principally,

- Feed preparation and analysis
- Acquisition of feedstock and catalysts
- Hydrotreating screening tests
- Detailed hydrotreating tests
- Products analyses

SRC's draft Subcontractor Report was reviewed by Scientific Authority and Stearns Catalytic Ltd. The approved final Subcontractor Report, which forms a part of this Project Report, is attached to this Chapter.

Key findings, conclusions and recommendations will be presented in Chapter IV.

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UPGRADING OF SYNTHETIC CRUDE DISTILLATES
WITH COMMERCIAL HYDROTREATING CATALYSTS

DSS File No. 06SQ.23440-4-9318

Subcontractor Report

Prepared By

D.W. Soveran
Saskatchewan Research Council
Petroleum Division

October 1986

SUMMARY

The objective of this project is to establish an independent Canadian capability in screening catalysts and synthetic crude feedstocks for hydro treating applications. This was achieved with this project by successfully testing five commercial hydrotreating catalysts using a partially upgraded gas oil feedstock.

A bench scale hydroprocessing unit was utilized to test the hydro treating effectiveness of five typical catalysts. Product qualities and yields were measured during the experimental program. Hydrogen consumption and the effect of various operating parameters (such as temperature, pressure and space velocity) were also determined.

Each catalyst was tested in a screening phase at a variety of operating conditions for a minimum of 120 hours (on-stream time). Product qualities were compared to determine the most effective catalyst, Shell S-424. This catalyst was then subjected to a long-term test (670 hours) to further evaluate its hydrotreating capabilities. The long-term test provided insufficient information regarding catalyst aging characteristics.

Two gas oil products from the long-term test were then tested in a Fluid Catalytic Cracking Microactivity Test unit to determine conversion to gasoline characteristics. The gas oils converted to gasoline at greater levels than conventional gas oils.

ACKNOWLEDGEMENTS

The Saskatchewan Research Council would like to thank the following suppliers for providing the samples of catalyst and the technical advice and information for the project:

1. American Cyanamid Ltd., Mississauga, Ontario
2. Akzo Chemie Ketjen Catalysts, Houston, Texas
3. Shell Catalysts, Houston, Texas
4. Halder-Topsoe, Inc., Houston, Texas.

PROJECT TEAM

The project team is as follows:

- | | |
|----------------|-------------------------|
| D.W. Soveran | - Project Leader |
| R. Ranganathan | - Project Advisor |
| N. Shatilla | - Research Technologist |
| C. Jackson | - Research Technologist |
| E. Ogu | - Research Engineer |

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I. INTRODUCTION

1.1 Project Objectives

The prime objective of this project is to develop a Canadian capability in screening catalysts and synthetic crude feedstocks for hydrotreating applications. This was to be accomplished by completing a number of specific project objectives, including:

1. Conduct hydrotreating experiments on synthetic crude distillates and evaluate the applicability of conventional hydrotreating technology to the refining of the new generation of feedstocks such as those derived from heavy oils and EOR (enhanced oil recovery) crudes.
2. Develop test procedures to secure reliable information and establish techniques for predicting performance in commercial hydrotreating units.
3. Obtain data, such as distillate quality, yield, etc., and qualitatively assess the ability to meet further downstream refining (e.g. catalytic cracking, reforming, visbreaking, etc.).
4. Develop Canadian expertise in the areas of hydrotreating of distillates derived from Canadian crude feedstocks.

5. Encourage the evolution of a distillate hydrotreating testing centre, readily accessible to potential customers from all sectors, public, private and universities.

1.2 Background

Conventional high quality crude production is declining in Canada and synthetic crude (from upgrading tar sand bitumen and other heavy feedstocks) production is increasing. Production expansion at Syncrude (Fort McMurray) and the completion of the Co-op upgrader (Regina) will significantly increase synthetic crude production in Canada. If further expansion of Syncrude and the Husky upgrader proceed, synthetic crude will increase even more in the near future.

Synthetic crudes differ significantly from naturally occurring crudes. Such characteristics as higher sulphur, nitrogen and aromatic levels make them more difficult to process with existing refining technology. It is essential to test conventional hydrotreating technology to establish its applicability, modify or develop new ones where necessary and develop experimental techniques to correlate the performance of syncrude distillates in conventional hydrotreaters.

The experimental program was divided into two segments, screening tests and a long-term test. Five commercially available catalysts were selected in consultation with the scientific authority. The feedstock

chosen was heavy coker gas oil from the Suncor bitumen upgrading plant in Fort McMurray. It was selected because it contained high levels of sulphur, nitrogen, aromatics and unsaturated compounds.

Conditions for the screening program were selected on the basis of known commercial hydrotreating operating parameters. One parameter (pressure) was maintained at one level suitable for this feedstock and another level not usually used in commercial operations. It was hoped that operating at a significantly lower pressure would help highlight the hydrotreating efficiency for a particular catalyst.

After completing the screening experiments, the liquid qualities were compared along with the product yields and the hydrogen consumption to determine the most effective catalyst. The data was also evaluated to determine the suitability of the chosen reaction conditions. The results indicated that the temperature range (360°-380°C) was not sufficient in all cases; a wider temperature range was selected for the long-term test (360°-390°C) to give a better idea of the optimum conditions.

The experimental design for the screening test was relatively simple; experiments were started with the lowest severity operation, succeeding experiments with higher severity. To cover the full range of conditions, eighteen (18) experiments were required.

Experimental design for the long-term test was modified so that

equivalent data could be gathered from fewer (but longer) experiments. The experiments were set in a similar manner so that the program proceeded with increasingly severe conditions. A mid-point set of conditions was placed strategically in the program to monitor catalyst aging.

From the onset of the experiment design it was expected that nitrogen removal and aromatic reduction would be the most difficult to achieve. Commercial hydrotreating of coker gas oils has shown denitrogenation to be the controlling parameter during approximately half of the catalyst's life. Desulphurization becomes the controlling parameter afterwards. Satisfactory aromatic reduction is difficult to achieve without increasing severity beyond that necessitated by sulphur and nitrogen removal. Several refiners have complained in the literature that aromatic levels in Canadian synthetic crude from Fort McMurray are too high for some typical petroleum products such as diesel fuel without additional processing. There are no specifications for aromatic levels but as synthetic crude becomes a larger fraction of total crude production it is expected that maximum levels will be set.

II. EXPERIMENTAL APPARATUS

The bench scale equipment used for the project was based on a unit obtained from Chemical Data Systems, Oxford, Pennsylvania. The unit was initially intended to process heavy resid feedstocks. The original gear pump was not suitable for gas oil feedstocks so it was replaced with a Milton-Roy adjustable stroke pump. Also added to the unit were load cells on the feed and product tanks so that those rates could be monitored constantly.

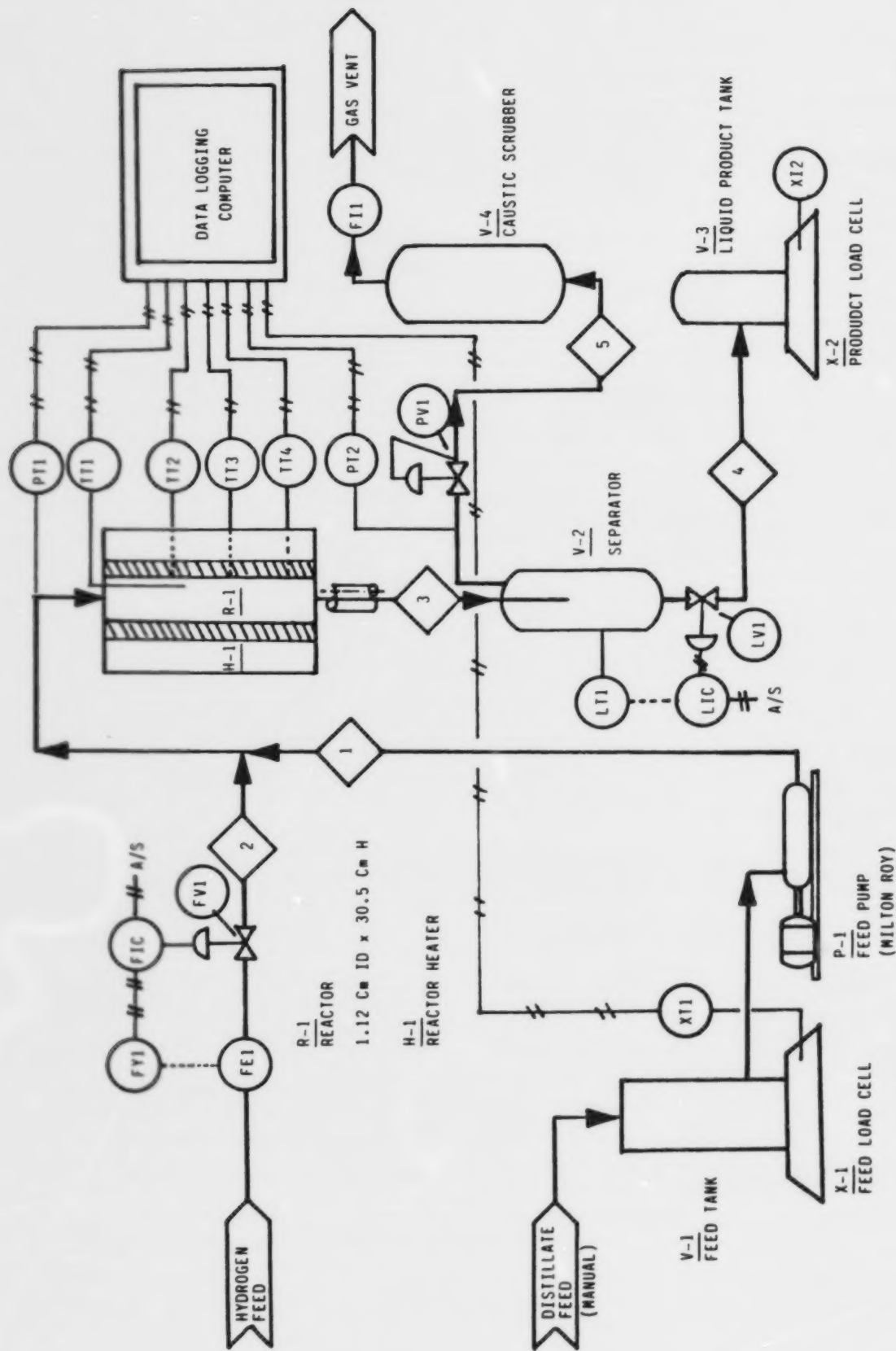
Operation of the unit is monitored with a computerized data acquisition system. Four temperatures, reactor inlet and outlet pressures, and the feed tank load cell are logged on the system.

Figure 1 is a schematic of the unit. The reactor is 1.27 cm I.D. by 30.5 cm long. The oil and hydrogen are mixed and then fed downflow over the catalyst bed. Gas flow is monitored and controlled by a mass flow control unit. Liquid feed is controlled by monitoring the change in weight of the feed tank and then adjusting the pump stroke to achieve the desired rate of weight change. The reactor temperature is monitored by a single thermocouple approximately two-thirds into the catalyst bed. Pressure is controlled by manually adjusting a back pressure regulator.

The reactor products are separated in a gas liquid separator. The liquid level is sensed by a vibrating reed sensor that triggers the

operation of a liquid let-down valve. The sensor worked flawlessly during the experiments, but the let-down valve experienced occasional failures due to the corrosion of an internal spring.

FIGURE 1: EXPERIMENTAL HYDROTREATING SYSTEM
 DSS File: 0650.23440-4-931R
 Stearns Catalytic/SRC Project: 4046



III. EXPERIMENTAL CONDITIONS

There were two experimental series performed for the project, a screening series and a long-term test. Five different catalysts were tested at a variety of conditions for the screening series. The most effective catalyst, based on desulphurization and denitrogenation, was selected for a long-term test.

The feedstock for both series was Suncor heavy coker gas oil. Table 1 summarizes its qualities. It was chosen because it was a readily available synthetic gas oil. It contains significant amounts of sulphur, nitrogen and aromatics and it would be a good test for the catalysts.

Five typical commercial hydrotreating catalysts were tested in the screening tests. The list included:

1. American Cyanamid HDS-20 - CoMo trilobe
2. Ketjen KF-840 - NiMo trilobe
3. Shell S-424 - NiMo tetralobe
4. Halder-Topsoe TK-581 - NiMo cylinders
5. American Cyanamid HDN-30 - NiMo trilobe

All are typical high activity hydrotreating catalysts. The operating conditions for the screening series was selected in consultation with the scientific authority and the prime contractor (Stearns Catalytic). The conditions were as follows:

Table 1A - Suncor Coker Gas Oil Analysis

Sample Description : Suncor Coker Gas Oil
Continuous Unit, Run No.: Feedstock
Date Reported : January 31, 1986

Density @ 15°C : 960.5 kg/m³
Relative Density @15/15°C : 0.9614
API Gravity @15/15°C : 15.7

Elemental Analysis:

Carbon : 84.0
Hydrogen : 10.9
Nitrogen : 0.26
Sulfur : 3.77

Trace Metals, ppm

Iron : 1.96
Nickel : 0.10
Vanadium : 0.12

Toluene Insolubles, wt % : 0.08
Pentane (n-C₅) insolubles, wt % : 0.09
Ramsbottom Carbon Residue, wt % : 0.43
Basic Nitrogen, wt % : 0.1681

Table 1B - Simulated Distillation of Feedstock

Sample Description : Suncor Coker Gas Oil
 Date Reported : November 21, 1985
 A.S. Sample No. : 85-1451

Distillation Method: GC Simulated Distillation

<u>Vol. Fraction Distilled</u>	<u>Boiling Point (°C)</u>	<u>Vol. Fraction Distilled</u>	<u>Boiling Point (°C)</u>
IBP	176.0	0.55	389.0
0.05	272.0	0.60	399.0
0.10	295.0	0.65	410.0
0.15	309.0	0.70	421.0
0.20	321.0	0.75	433.0
0.25	332.0	0.80	444.0
0.30	342.0	0.85	458.0
0.35	351.0	0.90	474.0
0.40	360.0	0.95	497.0
0.45	370.0	1.00	541.0
0.50	379.0	FBP	

<u>Summary:</u>	<u>Boiling Range (°C)</u>	<u>Vol. Fraction</u>
	IBP - 204	<u>0.900</u>
	204 - 343	<u>29.60</u>
	343 - 525	<u>68.00</u>
	525 - 600	<u> </u>
	525 + Residue	<u>1.500</u>
	600 + Residue	<u> </u>

1. Temperature: 360°, 370°, 380°C
2. Pressure: 6.0, 10.5 MPa
3. LHSV (Liquid Hourly Space Velocity): 0.5, 0.75, 1.0 hr⁻¹
4. Hydrogen feed rate: 900 cm³/cm³ liquid feed at 15°C, 1 atm.

These conditions are similar to commercial hydrotreating. No product quality standards were set; the intent of the program was to evaluate the catalysts under the same conditions.

Eighteen experiments were necessary to test all the possible combinations of conditions. The first experiment was duplicated at the end of the series to check for any change in catalyst activity. Each set of conditions was maintained for a minimum of six hours. Total on-stream time was at least 120 hours.

Two experiments per day were completed for the first series. The unit was shut down each night and restarted the next day. Restarting daily caused some minor time-consuming complications. Consequently, the subsequent series were performed non-stop (except for long weekends) to reduce the time period to complete each series. One of the periods was overnight, unattended operation for 9-10 hours (instead of the usual 6 hours). Total on-stream time for the remaining series was 140 hours. This did not significantly affect the results.

The series was structured so that it proceeded from the lowest severity to the highest severity. For example, the first experiment was 360°C,

10.5 MPa and 1.0 LHSV while the last was 380°C, 6.0 MPa and 0.5 LHSV. A maximum amount of information could be derived from the operation should the catalyst become fouled at the higher severity.

Density, sulphur, basic nitrogen and total nitrogen analysis were performed on the screening liquid products. The gas products were analyzed for C₁-C₆ hydrocarbons plus N₂, CO, CO₂, O₂, H₂S and H₂. Basic nitrogen analysis was performed because the total nitrogen analysis performed using a Perkin Elmer elemental analyzer did not give adequate resolution.

Based on CANMET's experience the catalyst was sulphided using the gas oil feedstock at 380°C, 10.5 MPa and 1.0 LHSV for 8 hours. It was felt this would be sufficient, but the results of the screening tests and the long-term test (where the presulphiding procedure was changed) indicated that it was not.

Initially the long-term test was planned to utilize similar (but fewer) conditions from the screening tests. The prime difference was to be the longer on-stream time (672 hours). After reviewing the screening data, some experiment design techniques¹ were utilized to obtain similar data with fewer experiments. The design sets of experiment used combinations of the minimums and maximums of the factor variables (temperature, pressure and space velocity). A single mid-point set of conditions is used at the start, middle and end of the series to provide a measure of catalyst

¹Murphy, T.D. Chemical Engineering (June 6, 1977) 168-82.

deactivation. The maximum reactor temperature was increased to 390°C because some of the screening results indicated that differences of 10°C were insufficient to change the product qualities significantly.

The conditions for the long-term test were:

1. Temperature: 360°, 390°C
2. Pressure: 6.0, 10.5 MPa
3. LHSV: 0.5, 1.0 hr⁻¹
4. Mid-point: 375°C, 8.75 MPa, 0.75 LHSV
5. Hydrogen feed rate: 900 cm³/cm³ liquid feed.

A total of eleven experiments were run during the long-term tests. With the exception of the mid-point conditions each set was maintained for 72 hours. The mid-points were maintained for 48, 24 and 24 hours at the beginning, middle and end of the series.

The liquid products were analyzed for the same characteristics as the screening series except only a total nitrogen analysis was performed. A new nitrogen analyzer was received and commissioned between the two series so a higher resolution nitrogen determination could be obtained. Gas analysis was similar to the screening series.

Liquid and gas products were measured to determine yields and hydrogen consumption.

Liquid samples were taken from the product receiver which was maintained at or near atmospheric pressure. Any dissolved gases were vented without being measured for most of the experiments. In a limited number of experiments the dissolved gas was measured with a wet test meter. It was less than 0.3% of the total gas production. The product receiver was then vented into the product gas line, but it was felt that little or no gas actually flowed because a slight pressure buildup (2-5 psig) would occur in the receiver during the course of an experiment. This pressure was insufficient to overcome line losses and the head pressure of the gas scrubber and wet test meter.

The liquid product from the first hour of a run was measured then discarded. The product for the last five hours was collected and submitted for analysis. Gas samples indicated that steady state operation was achieved after about an hour of operation. The samples showed that gas composition was relatively constant after the first hour of operation.

Gas samples were taken in a gas tight syringe on the low pressure side of the back pressure regulator before the gas scrubber. The sample was then injected into an off-line GC (gas chromatograph) for analysis.

As with the screening tests, the liquid product from the first hour of new operating conditions was collected, weighed and then discarded. The liquid products were then collected every 24 hours and submitted for analysis. The daily samples were then combined to form a composite sample

for the particular set of conditions and submitted for a detailed analysis. Gas was sampled 3 or 4 times per 24 hour period; the analyses were then averaged for that 24 hour period.

Results from the screening tests indicated that the sulphiding technique was not sufficient to adequately sulphide the catalyst. A technique supplied by Ketjen Catalysts was utilized for the long-term test. Their procedure was modified to use CS_2 (carbon disulphide) instead of DMDS (dimethyl disulphide). This procedure, given in Appendix C, resulted in a higher initial activity.

The three main variables of the experiments were reactor temperature, pressure and space velocity (LHSV). The temperature was controlled indirectly by controlling three external skin temperatures to achieve the desired bed temperature. This resulted in a small fluctuation of $\pm 1^\circ\text{C}$ of the bed temperature. The pressure was controlled with a manual back pressure regulator (BPR). Although the BPR was installed in a thermostatically-controlled block, the pressure control was sensitive to the ambient air temperature in the laboratory resulting in a pressure variation of $\pm 2\%$ of the setting. The LHSV was set by adjusting the stroke on the feed pump. The pump proved to be sensitive to the amount of liquid head-on the suction side. After the first few experiments, periodic adjustments could be made so that the desired LHSV was maintained.

IV. DISCUSSION OF RESULTS

4.1 Screening Tests

All the tested catalysts hydrotreated the feedstock with relative ease. Desulphurization was in the range of 92-98% removal. The reduction of basic nitrogen was usually greater than 85%. The density was also improved through hydrogenation and conversion (relatively small).

Table 2 gives a summary of the liquid product qualities for the Shell S-424 catalyst. Results for the other catalysts are given in Appendix A. Graphical representation of the results is also provided in Appendix B.

The sulphur levels in the product range from 0.04% to 0.3%. A sulphur level of 0.3% (maximum) is usually the specification for material with this boiling range. All of the catalysts provided this level of removal, even at the lower operating pressures.

Liquid product basic nitrogen values ranged from 0 to .0300% (wt). A maximum of 0.0300% (wt) is considered suitable for fluid catalytic cracking (FCC) feedstocks. Basic nitrogen will deactivate FCC catalysts because the nitrogen neutralizes the acid sites of the catalysts.

The total nitrogen values for the screening tests cannot be considered

Table 2 - Liquid Product Qualities Shell S-424 Screening Test

CATALYST: SHELL S-424						
CONDITIONS			Density (kg/m ³)	S (wt %)	Basic N (wt %)	N (wt %)
P (MPa)	T (°T)	LHSV (hr ⁻¹)				
10.5	360	1.0	894.1	0.305	0.0050	0.18
10.5	370	1.0	891.2	0.211	0.0072	0.11
10.5	380	1.0	876.9	0.178	0.0002	0.08
10.5	360	0.75	883.2	0.113	0.0004	0.05
10.5	370	0.75	876.8	0.115	0.0009	0.02
10.5	380	0.75	869.7	0.144	0.0001	0.02
10.5	360	0.5	874.2	0.112	0.0001	0.04
10.5	370	0.5	869.3	0.067	0.0001	0.04
10.5	380	0.5	858.7	0.068	N.D.	0.01
6.0	360	1.0	901.8	0.134	0.0231	0.02
6.0	370	1.0	902.7	0.173	0.0188	0.02
6.0	380	1.0	898.9	0.112	0.0084	0.02
6.0	360	0.75	902.6	0.220	0.0158	0.02
6.0	370	0.75	897.2	0.137	0.0055	0.02
6.0	380	0.75	890.9	0.080	0.0018	0.02
6.0	360	0.5	895.9	0.128	0.0029	0.01
6.0	370	0.5	891.6	0.075	0.0020	0.03
6.0	380	0.5	885.3	0.081	0.0015	0.07
10.5	360	1.0	898.6	0.140	0.0105	0.38

reliable because of the analytical method used. The elemental analyzer gives appropriate ratios of carbon, hydrogen and nitrogen but absolute values (especially for low concentrations) are difficult to perform accurately.

Density of the product was reduced significantly from 960 kg/m³ to 900 kg/m³ primarily by hydrogenation of aromatic compounds. Some density reduction occurred due to conversion to lower boiling compounds but conversion at these conditions is small.

There are several trends common to all the catalysts that can be determined from the product analysis. Lower LHSV's improved nitrogen and sulphur removal plus hydrogenation. The higher hydrogen pressure had higher levels of desulphurization, denitrogenation and hydrogenation. Higher temperatures improved the sulphur removal to a limited extent. There were several occasions where sulphur removal did not increase going from 370°C to 380°C. Denitrogenation and hydrogenation increased with higher reaction temperatures.

Higher temperature and lower LHSV's increased the methane (CH₄) to ethane (C₂H₆) ratios. No other changes in gas composition could be determined from the changes in the operating parameters.

A mass balance was performed for each experimental period. Liquid and gas yields as well as hydrogen consumption data were generated from the

mass balances. Table 3 summarizes the mass balance for the Shell S-424 catalyst. The mass balances for the other catalysts are given in Appendix A.

As with the product qualities there are several general trends that can be attributed to all the catalysts. Gas yield increased with increasing temperature, lower LHSV and lower hydrogen partial pressure. Only one catalyst, the Halder-Topsoe TK-581, maintained the same gas yield at the two pressures. With the Ketjen catalyst the gas yield increased more at the lower pressure than the other catalysts. As expected, hydrogen consumption was higher at the higher pressure and lower LHSV's. Increasing the reaction temperature increased the consumption only marginally in most cases.

The hydrogen consumption data should be evaluated from a qualitative perspective only. There are a number of sources of error in the determination of the consumption data. The hydrogen mass flowmeter has a range of 0 to 5 L/min. For this project the meter was operated in the .250 to .400 L/min range. The accuracy of the meter is $\pm 0.1\%$ of full scale or 0.005 L/min. The resulting error could be as much as 2% on hydrogen flow alone. There will be additional errors in the wet test meter. Another source is inevitable, small hydrogen leaks that occur with small systems. Consumption values, therefore, will contain several errors making the absolute values inconsistent with other researchers and commercial experiences.

Table 3 - Material Balance Shell S-424 Catalyst Screening Test

CATALYST: SHELL S-424							
CONDITIONS			Actual LHSV (hr ⁻¹)	Liquid Yield *(wt %FF)	Gas Yield *(wt %FF)	H ₂ Csump (g mol /g FF)**	Duration (h)
P (MPa)	T (°C)	LHSV (hr ⁻¹)					
10.5	360	1.0	1.06	97.2	4.3	0.0114	5.92
10.5	370	1.0	0.87	96.0	3.1	0.0235	9.67
10.5	380	1.0	0.98	96.2	5.8	0.0127	6.00
10.5	360	0.75	0.70	96.4	5.7	0.0133	6.00
10.5	370	0.75	0.72	95.1	5.5	0.0138	10.00
10.5	380	0.75	0.76	92.7	7.8	0.0140	6.00
10.5	360	0.5	0.49	95.8	6.4	0.0129	6.50
10.5	370	0.5	0.50	95.5	7.1	0.0138	10.67
10.5	380	0.5	0.50	96.2	9.4	0.0155	6.00
6.0	360	1.0	0.98	95.7	5.2	0.0098	6.25
6.0	370	1.0	1.01	96.4	5.7	0.0098	11.83
6.0	380	1.0	1.01	95.4	6.3	0.0096	6.00
6.0	360	0.75	0.74	95.6	5.3	0.0091	6.00
6.0	370	0.75	0.73	94.1	6.0	0.0073	6.00
6.0	380	0.75	0.73	93.5	7.8	0.0100	10.50
6.0	360	0.5	0.58	86.9	5.2	0.0084	6.00
6.0	370	0.5	0.51	94.6	7.0	0.0125	6.25
6.0	380	0.5	0.51	93.6	8.4	0.0128	10.75
10.5	360	1.0	1.00	95.3	8.3	0.0115	6.00
Total Time (h) = 142.34							

*wt % of fresh feed

**g mol H₂/g fresh feed

Another method for calculating hydrogen consumption would be to perform a hydrogen balance using the elemental analysis and the gas analysis. This was performed for one of the Shell runs. For conditions of 360°C, 10.5 mPa and 0.5 LHSV, the hydrogen consumption was 0.0129 g mol/g feed using the flowmeters only. Using the elemental analysis of the liquid feed and product plus the gas volume from the wet test and the gas analysis the consumption was 0.0106 g mol/g feed. The difference between the two is almost 18%. This has its errors too because the elemental analyzer provides reliable element ratios but absolute values can sometimes be suspect.

Hydrogen consumption data for the Ketjen KF-840 catalyst were significantly higher than the other catalysts at the 10.5 MPa operating pressure. Also the KF-840 was not the best hydrogenating catalyst. It was suspected that a leak was causing the loss of hydrogen. Several attempts were made to locate the leaks but none were found. Consumption was more comparable for the low pressure runs; no changes were made to the unit between the high and low pressure operation.

The catalysts can be rated on the individual measures of hydrotreating performance, desulphurization, denitrogenation and hydrogenation. Based on desulphurization the ranking is:

1. Cyanamid HDS-20 - Best
2. Shell S-424
3. Cyanamid HDN-30
4. Ketjen KF-840
5. Halder-Topsoe TK-581 - Worst

The HDS-20 can be expected to be the best because it is designed to be a high activity desulphurization catalyst. The HDN-30 is only marginally better than the KF-840 while the TK-581 is significantly poorer than the others.

For denitrogenation the ranking is slightly different:

- | | |
|-------------------------|---------|
| 1. Shell S-424 | - Best |
| 2. Ketjen KF-840 | |
| 3. Cyanamid HDS-20 | |
| 4. Halder-Topsoe TK-581 | |
| 5. Cyanamid HDN-30 | - Worst |

The ranking is based on the basic nitrogen analysis. The Shell catalyst is significantly better than the other catalysts. In one sample period it reduced the basic nitrogen below the detectable limits of the measuring device. A basic nitrogen specification for FCC feedstocks is usually set at 300 ppm (maximum) by most process licensors and catalyst manufacturers. There is some evidence that indicates that certain basic nitrogen compounds cause more FCC catalyst deactivation than others but that is not well defined at this time. At the lower pressure it was able to maintain basic nitrogen levels below significant levels (300 ppm); most of the other catalysts were unable to do so. With the exception of the HDS-20 all the catalysts are NiMo activated catalysts which should have made them excellent nitrogen removers. It is somewhat surprising that the HDS-20 finished a credible third in the ranking.

And for hydrogenation effectiveness the ranking is:

1. Shell S-424 - Best
2. Ketjen KF-840
3. Halder-Topsoe TK-581
Cyanamid HDS-20
4. Cyanamid HDN-30 - Worst

Hydrogenation is based on the density analysis of the liquid products. Other studies have shown that increasing aromatic content correlates to increased density. It is assumed that any conversion to lower boiling materials due to thermal effects will be the same for all catalysts. Figure 2 shows the true boiling point (TBP) curves, determined by simulated distillation, for the gas oil feedstock and two liquid products. One liquid product was from an HDS-20 experiment with conditions of 360°C, 10.5 MPa and 0.5 LHSV. The other liquid product was from a Shell S-424 run with the same conditions. The density for the HDS-20 product was 886.7 kg/m³ while the Shell product was 874.2 kg/m³. Some of the density difference may be due to the slight differences in the boiling ranges; some will also be due to aromatic contents.

Another interesting ranking for the catalysts is based on hydrogen consumption:

1. Ketjen KF-840 - Highest
2. Shell S-424
3. Cyanamid HDS-20
4. Cyanamid HDN-30
3. Halder-Topsoe TK-581 - Lowest

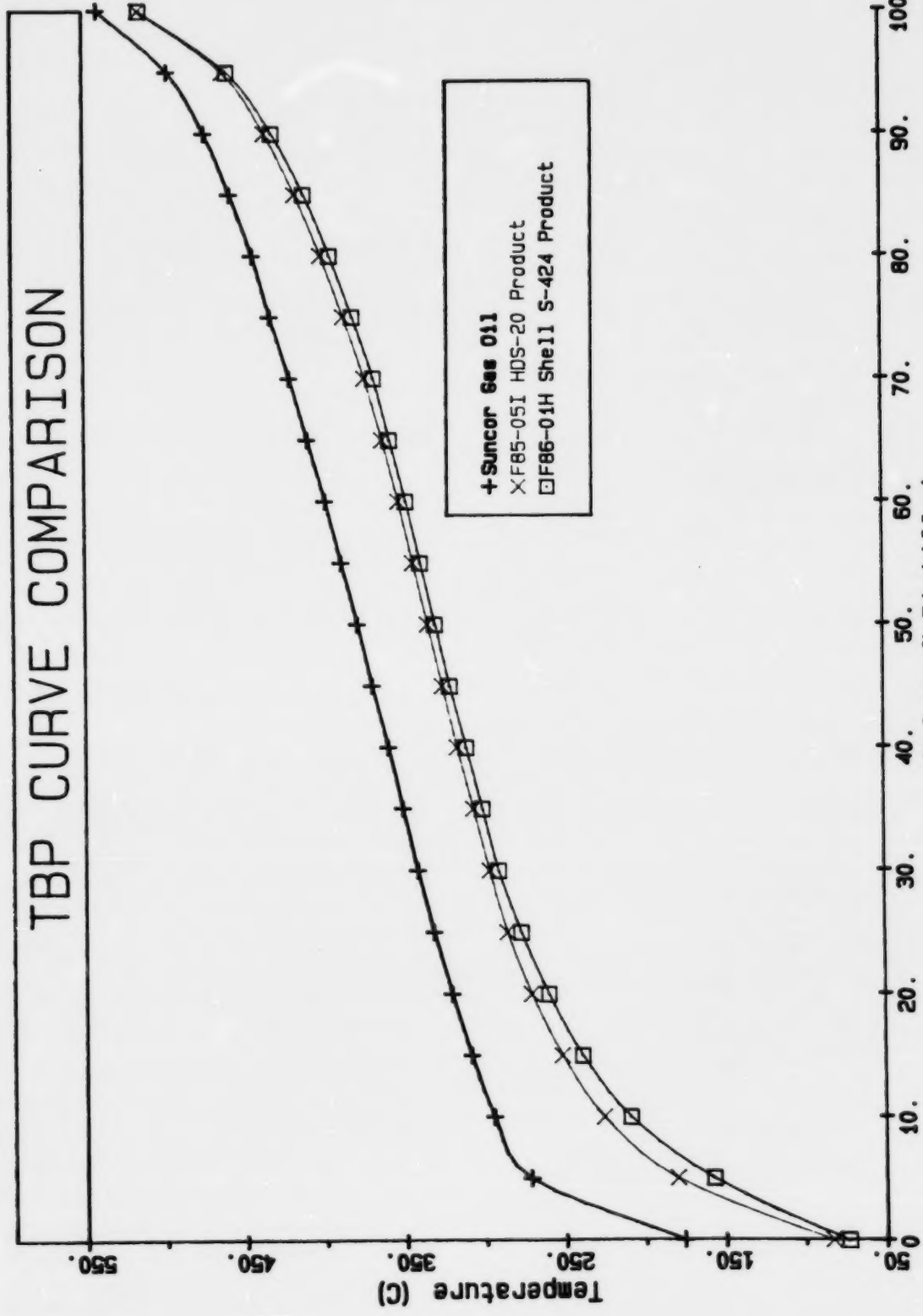


Figure 2 - TBP Curve Comparisons

This is based on the high pressure series only. One may expect that given the hydrogenation ranking, the consumption ranking would be a similar order (i.e. the best hydrogenation catalyst would have the highest consumption). A different order indicates that some of the hydrogen may be used to saturate non-aromatic compounds. This gives an indication of the selective nature of a particular catalyst.

Ranking hydrogen consumption for low pressure operation produces a different result:

- | | |
|-------------------------|------------|
| 1. Ketjen KF-840 | - Greatest |
| 2. Halder-Topsoe TK-581 | |
| 3. Cyanamid HDS-20 | |
| 4. Shell S-424 | |
| 5. Cyanamid HDN-30 | - Least |

The ranking of the Shell catalyst dropped significantly under the low pressure evaluation, yet its products consistently had the lowest density at both operating pressures.

Most of the catalysts exhibited no deactivation over the duration of the screening tests. Comparing the results of the first and last experiment actually indicated an improvement in desulphurization activity. This indicates that the catalysts were not completely sulphided. Some loss in denitrogenation activity could be noticed for some of the catalysts but it

was almost insignificant. Densities for the two experiments were within experimental error limits so no evaluation of hydrogenation activity could be made.

There was an attempt to identify sulphur compound types in a number of liquid samples. The samples were washed with certain reagents to remove certain sulphur compound types (such as mercury to remove elemental sulphur). This was unsuccessful because SRC's sulphur analyzer could not repeatedly and accurately measure the remaining sulphur.

The Shell S-424 ranked near or at the top of most categories and clearly was the best overall catalyst. It was recommended and accepted by the Scientific Authority for use in the long-term test.

A limited number of mechanical problems occurred during the screening tests that had little or no impact on the results. The first one was periodic plugging of the gas outlet line of the separator with ammonium salts. This occurred during the first HDS-20 run (the first catalyst tested). The line was heat traced and insulated and no more plugging was encountered. The second problem recurred several times. A spring in the liquid let down valve would eventually break due to either corrosion or hydrogen embrittlement of the spring. A spare valve was installed on the run with no impact on the operation of the unit.

4.2 Long-Term Test

The experimental conditions were modified for the long-term test to yield the same kind of data with fewer experiments. On-stream time was expected to be 28 days or 672 hours. The screening tests indicated that the presulphiding technique did not adequately sulphide the catalyst. A technique supplied by Ketjen Catalysts was modified and used. Carbon disulphide (CS_2) was added to the gas oil feedstock as the supply of sulphur for the catalyst.

A new nitrogen analyzer was received and commissioned by SRC at the start of the runs. Basic nitrogen analysis was no longer necessary to provide an accurate nitrogen determination.

At commercial upgrading facilities, a nitrogen level of 1100 ppm is considered the maximum for a synthetic gas oil product. With the exception of one experiment that level was easily achieved during the testing.

Sulphur levels were held to below 0.3% wt. except for one run.

The presulphiding technique appeared to make the catalyst very active initially. Some deactivation occurred early in the run and then it levelled out so that no more deactivation occurred.

Similar observations made with the short-term series could be made of

the long-term series. Denitrogenation, desulphurization and hydrogenation improved with increasing temperature and pressure. Lower LHSV's also improved product quality. Summaries of the composite liquid sample are provided in Tables 4 and 5; the data for the daily samples are provided in Appendix A. Graphical representation of the long-term test is also provided in Appendix B.

The catalyst performed adequately at the lowest pressure (6.0 MPa), but its performance was much better at the higher pressures. Higher pressures would probably extend its life as well.

A number of mechanical problems were encountered during the test. Several problems with the two gas chromatographs used during the long-term test were experienced. Electrical spikes and a leaking hydrogen transfer tube caused the loss of several analyses on one GC. The other GC was not intended to provide the analysis required by the hydrotreating operation. Its detector's response to H_2S was highly non-linear making calibration impossible. The result is that its analyses were adjusted with the help of the analyses from the screening tests. The mass balance for the long-term test has more error than the screening tests mass balance.

The mass balance data are provided in Table 6. The daily material balances are provided in Appendix A. Similar hydrogen consumption and product qualities were obtained for the screening and the extended experiments.

**Table 4 - Liquid Product Qualities Shell S-424
Catalyst Long-Term Test**

CONDITIONS				COMPOSITE				
Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)	Period #	Density (kg/m ³)	Sulphur (wt %)	Nitrogen (wppm)	Carbon (wt %)	Hydrogen (wt %)
375	0.75	8.75	F86-07B	863.5	0.06	4.9	86.6	12.7
360	1.0	10.5	F86-07C	887.0	0.03	75.1	86.4	12.7
390	1.0	10.5	F86-07D	878.3	0.01	39.0	86.0	12.7
360	0.5	10.5	F86-07E	900.1	0.14	214.0	86.0	12.5
390	0.5	10.5	F86-07F	876.0	0.05	6.8	86.0	13.0
375	0.75	8.75	F86-07G	902.8	0.18	400.0		
360	1.0	6.0	F86-07H	915.4	0.46	1158.0	86.4	11.9
390	1.0	6.0	F86-07I	903.4	0.11	502.0	85.7	12.1
360	0.5	6.0	F86-07J	909.1	0.22	706.0	86.2	12.2
390	0.5	6.0	F86-07K	889.0	0.06	69.4	86.4	12.5
375	0.75	8.75	F86-07L	901.9	0.20	317.0		

**Table 5 - Composite Liquid Product Distillations Shell S-424
Catalyst Long-Term Test**

CONDITIONS				SIM. DIST. SUMMARY (COMPOSITE SAMPLE)			
Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)	Period #	IBP-204 vol %	204-343 vol %	343-525 vol %	525+ vol %
375	0.75	8.75	F86-07B	16.9	57.8	25.3	0.8
360	1.0	10.5	F86-07C	8.8	48.8	44.4	
390	1.0	10.5	F86-07D	10.3	51.9	37.8	
360	0.5	10.5	F86-07E	2.9	44.5	52.6	
390	0.5	10.5	F86-07F	8.0	50.4	41.6	
375	0.75	8.75	F86-07G				
360	1.0	6.0	F86-07H	2.0	40.0	57.2	
390	1.0	6.0	F86-07I	3.7	44.1	52.2	
360	0.5	6.0	F86-07J	2.4	42.2	55.4	
390	0.5	6.0	F86-07K	6.5	48.6	44.9	
375	0.75	8.75	F86-07L				

Table 6 - Material Balance Shell S-424 Catalyst Long-Term Test

Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)	Period #	Actual LHSV	Total Liq Prod (wt % FF)	Gas Prod (wt % FF)	Time (hr)	H ₂ Consumed (gmol/gF)
375	0.75	8.75	F86-07B	0.76	96.2	6.6	48.4	0.0136
360	1.0	10.5	F86-07C	0.98	94.4	4.2	71.7	0.0122
390	1.0	10.5	F86-07D	1.04	90.5	4.3	69.5	0.0136
360	0.5	10.5	F86-07E	0.50	96.8	3.3	73.6	0.0109
390	0.5	10.5	F86-07F	0.50	95.8	3.9	72.0	0.0134
375	0.75	8.75	F86-07G	0.68	94.1	3.3	24.1	0.0222
360	1.0	6.0	F86-07H	1.00	96.7	3.9	72.0	0.0078
390	1.0	6.0	F86-07I	0.99	95.9	4.1	72.0	0.0100
360	0.5	6.0	F86-07J	0.51	95.9	4.3	72.1	0.0078
390	0.5	6.0	F86-07K	0.50	94.7	5.1	72.0	0.0108
375	0.75	8.75	F86-07L	0.76	96.4	3.3	24.0	0.0237

A unit fail-safe (unattended shut down) occurred during the F86-07D run when a filter on the gas outlet became plugged. After changing the filter, the unit was restarted the next day without any trouble. Liquid recovery for this experiment was lower than normal, 91.4% versus 95%. There is no logical explanation for this except that this was the only abnormal occurrence for that experiment.

After completing the F86-07D run the unit was shut down for a long weekend. The unit had run non-stop from the start of presulphiding to the end of F86-07D. During the restart one of the reactor heaters behaved erratically, sometimes overheating and sometimes not responding to changes in the set point. The cause was determined to be a faulty temperature sensor. The feed to the unit was started prior to reaching the desired temperature. Using two heaters it appeared the reactor was unable to be brought to the desired temperature. Ultimately a high pressure drop developed across the catalyst bed along with abnormally high skin temperatures. Finally the unit was shut down and the catalyst removed. Coke had formed around the internal thermowell preventing an accurate reading. Coke formation in the bed had caused the high pressure drop.

The reactor was filled with fresh catalyst and then presulphided with CS_2 spiked gas oil. The remaining experiments were completed non-stop without further problems. The second load of catalyst accumulated 504 hours of on-stream time.

By evaluating the mid-point experiments catalyst activity changes can be monitored. Extremely good desulphurization, denitrogenation and hydrogenation was achieved during the first experiment; the F86-07G run was actually 7 days after presulphiding, while the F86-07L was 20 days after. This indicates that initially the catalyst was very active, then rapid deactivation to a stable level. Further deactivation was not experienced with the long-term test.

The used catalyst from the 21-day period was submitted along with new catalyst to the University of Saskatchewan for pore size and surface area analysis by BET (Brunauer-Emmett-Teller). Figure 3 shows the pore size analysis for the new and used catalyst. The surface areas were 145 m²/g (corrected for carbon content) for the used and 155 m²/g for the new. Carbon content was 4.04 wt % for the catalyst. These results show that the catalyst had changed but probably not yet to a significant level.

4.3 Fluid Catalytic Cracking (FCC) Microactivity Test (MAT)

Two of the gas oil products from the long-term test were selected for FCC MAT tests to measure the conversion to gasoline of the synthetic gas oil. The samples selected represented the lowest (F86-07H) and the highest (F86-07F) severity of operation.

The samples were distilled in a short path distillation apparatus to remove materials boiling less than 315°C. There are significant amounts of

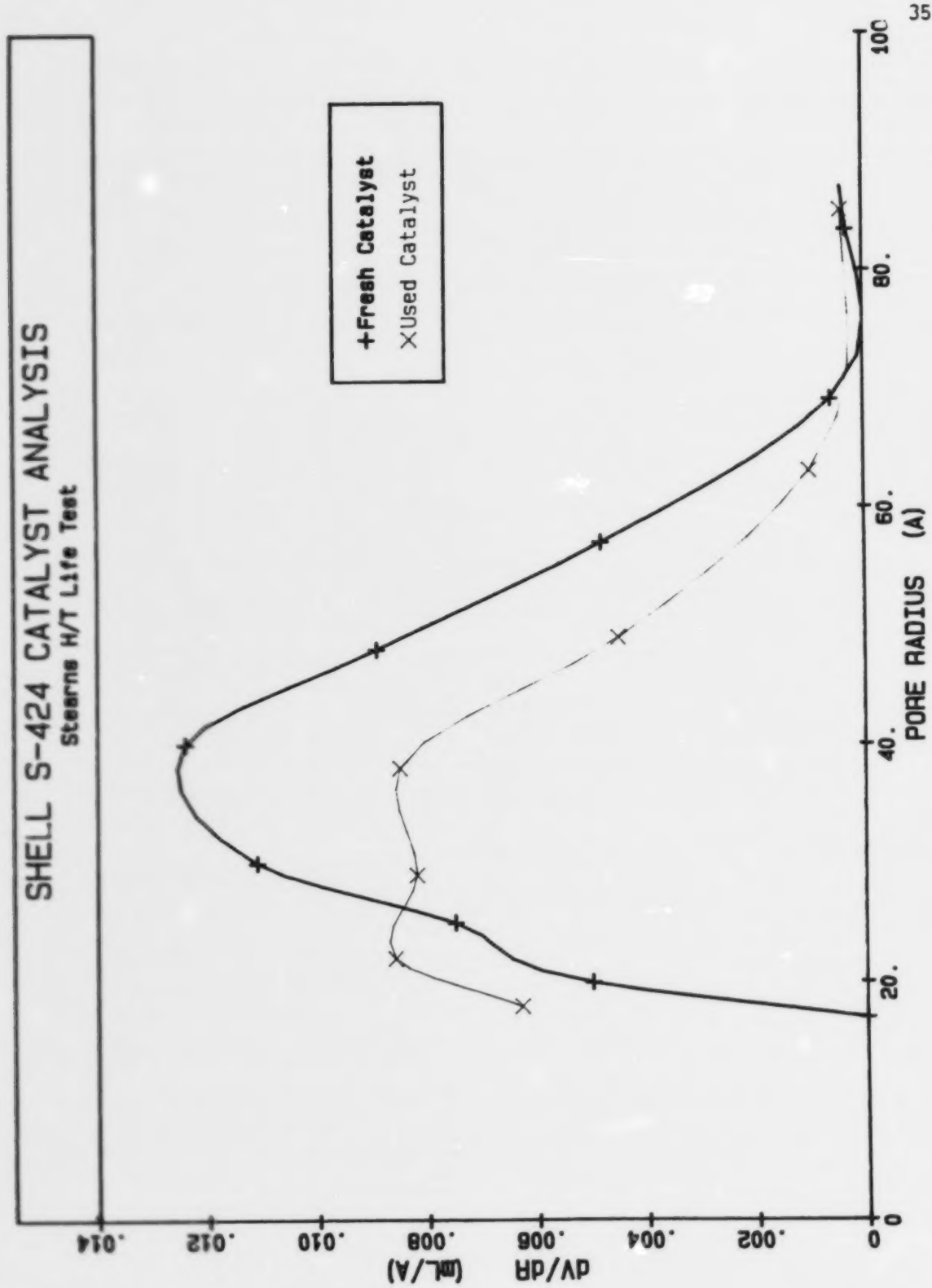


Figure 3 - Catalyst Analysis Long-Term Test

light material in the gas oil which can give misleading data about their conversion potential.

The MAT procedure was based on ASTM D-3907-80 with some minor modifications. The catalyst used was a steam-deactivated fresh catalyst, Engelhard HEZ-55.

The difference in conversion is mostly due to the feedstock quality, the 10% difference in the catalyst to oil ratio will result in a small difference in conversion (<1%). The coke yield was not determined but it is usually 4-6 wt % of the feed.

The MAT results show a dramatic difference between the two gas oil products. The most severely treated gas oil (F86-07F) had the highest conversion to gasoline. Also significant was the large gas make of this sample. The high conversions indicate that the aromatic content is quite low (since aromatics do not convert). A conventional gas oil (derived from a light conventional crude) would have a conversion in the 75% range with this catalyst.

The second sample (F86-07H) was closer in quality to conventional gas oil with a similar conversion and gas make. The high conversions indicate that the hydrotreating was more severe than necessary to produce a quality product. Hydrotreating conditions would have to be optimized to produce a suitable FCC feedstock.

Table 7 - FCC MAT Experiments

Sample Description	F86-07F	F86-07H
Conversion, wt %	91.0	81.3
Yields, wt % of Feed		
Gasoline, C ₆ -216°C	65.8	47.5
LCO, 216°-343°C	13.5	20.5
Bottoms, 343°C+	1.7	6.2
Gas	11.3	9.8
Catalyst: Engelhard HEZ-55, steam deactivated at 732°C for 1 hour		
Catalyst/Oil Ratio	3.30	3.01
Temperature	482°C	482°C
Injection Period	75 s	75 s

Conversion is a measure of the yield of material boiling below 216°C and is calculated in the following manner:

$$\text{Conversion, wt \%} = \frac{F - \frac{R \times L}{100} - H}{100} \times 100$$

F = grams feed
R = wt % liquid product boiling above 216°C
L = grams liquid product
H = grams liquid holdup at reactor exit

For sample F86-07F:

F = 1.21 g, R = 18.4 wt %, L = 0.59 g, H = 0.001 g (neglected).

V. CONCLUSIONS

The project provided significant hydrotreating information and helped to establish expertise for feedstock and catalyst screening using hydrotreating technology. The screening series was sufficient to identify quantifiable differences between the various catalysts. The results were consistent with a few exceptions, which can be expected with any experimental series. The long-term test also provided valuable information, plus it proved the equipment is capable of extended operation with minimal care.

The screening series proved that all the catalysts were capable of producing a quality, hydrotreated product. The series did show significant differences between the catalysts so that the best was easily identifiable. With better experimental design the number of experiments could have been reduced with the same quality information. The experimental period for an individual run should have been longer (minimum 8 hours) to produce more liquid product. The quantity of product was barely satisfactory (especially for the low LHSV's) to perform the analysis; an important consideration because some analyses were repeated when inconsistent results were noticed. The data may also indicate other analyses are necessary so a little extra product is beneficial.

The screening tests also highlighted the necessity of a proper presulphiding procedure. The gas oil alone, even though it contained a high sulphur level, was an insufficient source of sulphur for the catalyst.

With the proper presulphiding procedure used in the life test the catalyst activity was higher producing a better quality product. There was a small indication of deactivation in the screening tests while the long-term test showed almost none. Proper presulphiding may help delay deactivation of the catalyst.

The long-term test showed that longer on-stream times were necessary before aging could be discerned. The equipment could be operated for longer periods to determine deactivation characteristics.

The FCC MAT test is useful to help judge the characteristics of the hydrotreated product. It is a real measure of the feedstock conversion performance rather than one deduced from a variety of analyses. It is a cost-effective means for determining some important characteristics.

The quality of the distillate product from most of the experiments was within acceptable ranges for use as a feedstock in typical refining units. The MAT test showed that the material would convert readily to gasoline in an FCC unit. Total nitrogen content less than 1200 ppm and basic nitrogen less than 300 ppm are requirements for suitable FCC feedstocks. Sulphur levels less than 0.3 wt % are also required for FCC feedstocks. The Shell catalyst provided significant hydrogenation of aromatic compounds. Additional analysis would be necessary to determine if it was sufficient to provide a suitable base stock for distillate fuels such as diesel and jet fuel as well as a base stock for lubricating oils. There was no separation

made of the product to determine the quality of the naphtha fraction therefore no comments can be made on its suitability for downstream refining.

Future work that would enhance the development of expertise would be a true catalyst aging test. A run length of three months or more would be necessary to fully establish a deactivation curve for a particular catalyst. There is no reason to expect that the equipment is not capable of an extended run. Testing of newer catalysts and feedstocks that experienced different primary upgrading steps would add to the data base and maintain a current contact with other developments.

APPENDIX A

TABLES

**Table A-1 - Liquid Product Qualities Cyanamid HDS-20
Catalyst Screening Test**

CATALYST: CYANAMID HDS-20

P (MPa)	CONDITIONS		Density (kg/m ³)	S (wt %)	Basic N (wt %)	N (wt %)
	T (°C)	LHSV (hr ⁻¹)				
10.5	360	1.0	902.8	0.376	0.0320	0.07
10.5	370	1.0	895.2	0.042	0.0130	0.08
10.5	380	1.0	893.3	0.052	0.0094	
10.5	360	0.75	898.0	0.106	0.0060	0.10
10.5	370	0.75	891.6	0.087	0.0021	0.06
10.5	380	0.75	887.4	0.070	0.0039	0.12
10.5	360	0.5	886.7	0.083	0.0052	0.21
10.5	370	0.5	879.8	0.063	0.0021	0.23
10.5	380	0.5	884.6	0.076	0.0042	0.21
6.0	360	1.0	906.3	0.139	0.0494	0.28
6.0	370	1.0	903.2	0.085	0.0376	0.12
6.0	380	1.0	901.5	0.085	0.0277	0.10
6.0	360	0.75	904.2	0.176	0.0373	0.10
6.0	370	0.75	898.3	0.097	0.0183	0.16
6.0	380	0.75	894.6	0.086	0.0133	0.25
6.0	360	0.5	901.3	0.256	0.0073	0.27
6.0	370	0.5	895.9	0.157	0.0157	0.46
6.0	380	0.5	890.2	0.056	0.0078	0.13
10.5	360	1.0	902.0	0.131	0.0335	0.14

**Table A-2 - Liquid Product Qualities Ketjen KF-840
Catalyst Screening Test**

CATALYST: KETJEN KF-840

CONDITIONS			Density (kg/m ³)	S (wt %)	Basic N (wt %)	N (wt %)
P (MPa)	T (°C)	LHSV (hr ⁻¹)				
10.5	360	1.0	897.6	0.273	0.0096	0.35
10.5	370	1.0	891.1	0.178	0.0042	0.30
10.5	380	1.0	887.0	0.090	0.0013	0.20
10.5	360	0.75	890.8	0.181	0.0045	0.12
10.5	370	0.75	883.0	0.115	0.0024	0.13
10.5	380	0.75	871.0	0.177	0.0003	0.40
10.5	360	0.5	884.9	0.168	0.0013	0.40
10.5	370	0.5	897.3	0.162	0.0020	0.30
10.5	380	0.5	873.0	0.133	0.0010	0.04
6.0	360	1.0	905.0	0.267	0.0296	0.20
6.0	370	1.0	904.8	0.202	0.0247	0.20
6.0	380	1.0	900.6	0.128	0.0118	0.16
6.0	360	0.75	904.5	0.246	0.0206	0.30
6.0	370	0.75	900.0	0.123	0.0117	0.20
6.0	380	0.75	897.4	0.204	0.0050	0.09
6.0	360	0.5	900.5	0.195	0.0080	0.43
6.0	370	0.5	893.3	0.118	0.0040	0.36
6.0	380	0.5	887.9	0.106	0.0018	0.43
10.5	360	1.0	899.7	0.210	0.0114	0.44

**Table A-3 - Liquid Product Qualities Halder-Topsoe
TK-581 Catalyst Screening Test**

CATALYST: HALDER-TOPSOE TK-581

P (MPa)	CONDITIONS		Density (kg/m ³)	S (wt %)	Basic N (wt %)	N (wt %)
	T (°C)	LHSV (hr ⁻¹)				
10.5	360	1.0	902.5	0.293	0.0200	0.20
10.5	370	1.0	897.2	0.204	0.0080	0.25
10.5	380	1.0	891.4	0.118	0.0046	0.03
10.5	360	0.75	896.9	0.189	0.0079	0.06
10.5	370	0.75	889.9	0.083	0.0048	0.04
10.5	380	0.75	882.5	0.092	0.0010	0.04
10.5	360	0.5	888.7	0.145	0.0020	0.04
10.5	370	0.5	880.6	0.073	0.0020	0.05
10.5	380	0.5	871.5	0.064	0.0003	0.04
6.0	360	1.0	907.3	0.296	0.0556	0.04
6.0	370	1.0	908.4	0.301	0.0565	0.06
6.0	380	1.0	905.1	0.208	0.0375	0.02
6.0	360	0.75	906.7	0.359	0.0412	0.04
6.0	370	0.75	904.5	0.298	0.0306	0.03
6.0	380	0.75	899.6	0.201	0.0169	0.04
6.0	360	0.5	904.4	0.385	0.0248	0.07
6.0	370	0.5	899.4	0.227	0.0135	0.05
6.0	380	0.5	892.7	0.127	0.0068	0.06
10.5	360	1.0	904.5	0.254	0.0301	0.08

Table A-4 - Liquid Product Qualities Cyanamid HDN-30
Catalyst Screening Test

CATALYST: CYANAMID HDN-30

CONDITIONS		LHSV (hr ⁻¹)	Density (kg/m ³)	S (wt %)	Basic N (wt %)	N (wt %)
P (MPa)	T (°C)					
10.5	360	1.0	903.4	0.310	0.0315	0.08
10.5	370	1.0	899.1	0.194	0.0199	0.08
10.5	380	1.0	892.9	0.076	0.0129	0.04
10.5	360	0.75	897.6	0.194	0.0178	0.06
10.5	370	0.75	893.7	0.174	0.0107	0.06
10.5	380	0.75	885.4	0.034	0.0072	0.08
10.5	360	0.5	889.6	0.130	0.0053	0.09
10.5	370	0.5	884.6	0.108	0.0043	0.11
10.5	380	0.5	875.4	0.047	0.0041	0.04
6.0	360	1.0	909.5	0.351	0.0652	0.05
6.0	370	1.0	908.0	0.230	0.0514	0.08
6.0	380	1.0	904.4	0.156	0.0317	0.05
6.0	360	0.75	908.1	0.278	0.0471	0.05
6.0	370	0.75	904.2	0.171	0.0299	0.07
6.0	380	0.75	900.4	0.097	0.0173	0.07
6.0	360	0.5	904.4	0.254	0.0255	0.03
6.0	370	0.5	898.8	0.158	0.0132	0.06
6.0	380	0.5	892.8	0.032	0.0064	0.04
10.5	360	1.0	903.0	0.131	0.0214	0.04

Table A-5 - Gas Analysis Dynamid HDS-20 Catalyst Screening Test

CONDITIONS			(mol fractions)												
P (MPa)	T (°C)	LHSV (hr ⁻¹)	H ₂	C ₂ H ₆	C ₃ H ₈	i-C ₄	n-C ₄	i-C ₅	n-C ₅	C ₆ +	CO ₂	H ₂	N ₂	O ₂	H ₂ S
10.5	360	1.0	0.0025	0.0019	0.0014		0.0006	0.0001	0.0011	0.0014		0.9572	0.0040		0.0309
10.5	370	1.0	0.0029	0.0020	0.0017	0.0001	0.0007	0.0004	0.0011	0.0017		0.9577	0.0038		0.0279
10.5	380	1.0	0.0038	0.0027	0.0020	0.0001	0.0009	0.0002	0.0003	0.0030	0.0022	0.9518	0.0036		0.0295
10.5	360	0.7	0.0020	0.0012	0.0011	0.0001	0.0004	0.0001	0.0001	0.0015		0.9636	0.0006		0.0294
10.5	370	0.7	0.0042	0.0027	0.0022	0.0001	0.0009	0.0002	0.0004	0.0023		0.9556	0.0022		0.0292
10.5	380	0.7	0.0054	0.0034	0.0027	0.0002	0.0013	0.0004	0.0005	0.0027		0.9509	0.0028		0.0297
10.5	360	0.5	0.0064	0.0039	0.0030	0.0002	0.0014	0.0003	0.0005	0.0026		0.9452	0.0051	0.0003	0.0312
10.5	370	0.5	0.0066	0.0040	0.0020	0.0002	0.0015	0.0004	0.0006	0.0030		0.9503	0.0024		0.02910
10.5	380	0.5	0.0060	0.0043	0.0025	0.0003	0.0013	0.0003	0.0004	0.0026		0.9440	0.0068		0.0315
6.0	360	1.0	0.0020	0.0015	0.0012	0.0001	0.0005	0.0002	0.0002	0.0014		0.9622	0.0029		0.0278
6.0	370	1.0	0.0039	0.0026	0.0030	0.0001	0.0009	0.0002	0.0003	0.0021		0.9523	0.0045		0.0300
6.0	380	1.0	0.0051	0.0031	0.0026	0.0001	0.0011	0.0002	0.0005	0.0024		0.9513	0.0020		0.0316
6.0	360	0.7	0.0036	0.0024	0.0020	0.0001	0.0008	0.0002	0.0003	0.0020		0.9513	0.0050		0.0322
6.0	370	0.7	0.0070	0.0044	0.0036	0.0002	0.0016	0.0004	0.0006	0.0116		0.9246	0.0097		0.0363
6.0	380	0.7	0.0096	0.0053	0.0043	0.0003	0.0018	0.0004	0.0007	0.0087		0.9363	0.0009		0.0316
6.0	360	0.5	0.0039	0.0026	0.0021	0.0001	0.0009	0.0002	0.0004	0.0022		0.9503	0.0041		0.0332
6.0	370	0.5	0.0081	0.0048	0.0038	0.0002	0.0016	0.0005	0.0006	0.0038		0.9379	0.0043		0.0344
6.0	380	0.5	0.0121	0.0066	0.0053	0.0004	0.0023	0.0005	0.0009	0.0044		0.9319	0.0032		0.0324
10.5	360	1.0	0.0020	0.0014	0.0010		0.0004	0.0001	0.0002	0.0011		0.9578	0.0063		0.0297

Table A-6 - Gas Analysis Ketjen KF-840 Catalyst Screening Test

Table A-6 - Gas Analysis Ketjen KF-840 Catalyst Screening Test															
CONDITIONS		(mol fractions)													
P (MPa)	T (°C)	LHSV (hr ⁻¹)	H ₂	C ₂ H ₆	C ₃ H ₈	i-C ₄	n-C ₄	i-C ₅	n-C ₅	C ₆ ⁺	CO ₂	H ₂	N ₂	O ₂	H ₂ S
10.5	360	1.0	0.0026	0.0019	0.0017	0.0001	0.0007	0.0001	0.0004	0.0014		0.9525	0.0033		0.0355
10.5	370	1.0	0.004	0.0029	0.002	0.0001	0.001	0.0002	0.0003	0.0008		0.9484	0.0036		0.0367
10.5	380	1.0	0.0092	0.0042	0.0038	0.0003	0.0016	0.0003	0.0005	0.002		0.9376	0.0044		0.0362
10.5	360	0.7	0.0036	0.0026	0.0024	0.0001	0.001	0.0002	0.0003	0.0018		0.946	0.0025		0.0395
10.5	370	0.7	0.0071	0.0046	0.0036	0.0003	0.0017	0.0004	0.0005	0.0025		0.9329	0.0064		0.04
10.5	380	0.7	0.0125	0.0076	0.0061	0.0006	0.0026	0.0006	0.0008	0.0031		0.9228	0.033		0.0399
10.5	360	0.5	0.0071	0.0045	0.004	0.0002	0.0015	0.0003	0.0005	0.0026		0.9249	0.0021	0.0003	0.0523
10.5	370	0.5	0.0114	0.0068	0.0056	0.0004	0.0023	0.0005	0.0006	0.0031		0.9138	0.0049		0.0505
10.5	380	0.5	0.0196	0.0107	0.0081	0.0005	0.003	0.0006	0.0009	0.0027		0.8993	0.0033		0.0513
6.0	360	1.0	0.0017	0.0014	0.0009	0.0001	0.0005	0.0001	0.0002	0.0015		0.9615	0.0008		0.0313
6.0	370	1.0	0.0028	0.0026	0.0017	0.0001	0.0008	0.0002	0.0004	0.0019		0.9501	0.0065		0.0335
6.0	380	1.0	0.0052	0.0034	0.0028	0.0002	0.0012	0.0003	0.0005	0.0027		0.9412	0.0099		0.0326
6.0	360	0.7	0.0025	0.0019	0.0017	0.0001	0.0007	0.0003	0.0003	0.0017		0.9429	0.0134		0.0344
6.0	370	0.7	0.0047	0.0031	0.0027	0.0002	0.0012	0.0003	0.0004	0.0025		0.9379	0.013		0.0342
6.0	380	0.7	0.0084	0.0053	0.0042	0.0003	0.002	0.0005	0.0007	0.003		0.9179	0.0195		0.0382
6.0	360	0.5	0.0037	0.0026	0.0024	0.0001	0.001	0.0003	0.0005	0.0022		0.9363	0.0151		0.0359
6.0	370	0.5	0.0072	0.0045	0.0039	0.0003	0.001	0.0004	0.0007	0.0033		0.9272	0.0177		0.0331
6.0	380	0.5	0.012	0.0076	0.006	0.0006	0.0017	0.0007	0.0001	0.004		0.9159	0.0154		0.0347
10.5	360	1.0	0.0024	0.007	0.0017	0.0001	0.0026	0.0002	0.0003	0.0015		0.9433	0.0123		0.0357

Table A-7 - Gas Analysis Shell S-424 Catalyst Screening Test

CONDITIONS				(mol fractions)												
P (MPa)	T (°C)	WHSV (hr ⁻¹)	1) CH ₄	C ₂ H ₆	C ₃ H ₈	i-C ₄	n-C ₄	i-C ₅	n-C ₅	C ₆ +	CO ₂	H ₂	N ₂	O ₂	H ₂ S	
10.5	360	1.0	0.0026	0.0014	0.0016	0.0001	0.0006	0.0001	0.0002	0.0021		0.9624	0.0015		0.0275	
10.5	370	1.0	0.0052	0.0026	0.0033	0.0002	0.0012	0.0003	0.0003	0.0028		0.9468	0.0079		0.0294	
10.5	380	1.0	0.0065	0.0045	0.0033	0.0003	0.0017	0.0004	0.0005	0.0037		0.9471	0.0031		0.0289	
10.5	360	0.7	0.0034	0.0026	0.0032	0.0002	0.0011	0.0003	0.0005	0.0049		0.9530	0.0026		0.0283	
10.5	370	0.7	0.0052	0.0035	0.0006	0.0003	0.0015	0.0004	0.0006	0.0054		0.9511	0.0036		0.0278	
10.5	380	0.7	0.0092	0.0055	0.0061	0.0005	0.0022	0.0006	0.0008	0.0118		0.9316	0.0031		0.0285	
10.5	360	0.5	0.0050	0.0032	0.0045	0.0003	0.0014	0.0003	0.0005	0.0048		0.9430	0.0074		0.0296	
10.5	370	0.5	0.0072	0.0046	0.0055	0.0006	0.0020	0.0005	0.0007	0.0080		0.9364	0.0043		0.0303	
10.5	380	0.5	0.0125	0.0078	0.0098	0.0020	0.0035	0.0011	0.0016	0.0189		0.9105	0.0014		0.0309	
6.0	360	1.0	0.0024	0.0016	0.0018	0.0001	0.0006	0.0002	0.0003	0.0036		0.9576	0.0005		0.0314	
6.0	370	1.0	0.0037	0.0026	0.0030	0.0001	0.0010	0.0002	0.0004	0.0033		0.9507	0.0019		0.0330	
6.0	380	1.0	0.0060	0.0036	0.0043	0.0002	0.0015	0.0004	0.0005	0.0042		0.9468	0.0010		0.0316	
6.0	360	0.7	0.0030	0.0020	0.0024	0.0001	0.0008	0.0002	0.0003	0.0029		0.9560	0.0017		0.0305	
6.0	370	0.7	0.0051	0.0032	0.0038	0.0002	0.0012	0.0003	0.0006	0.0039		0.9513	0.0007		0.0297	
6.0	380	0.7	0.0087	0.0052	0.0057	0.0004	0.0020	0.0005	0.0008	0.0062		0.9391	0.0020		0.0295	
6.0	360	0.5	0.0051	0.0033	0.0039	0.0002	0.0014	0.0003	0.0004	0.0027		0.9478	0.0005		0.0344	
6.0	370	0.5	0.0083	0.0050	0.0058	0.0003	0.0020	0.0004	0.0007	0.0049		0.9381	0.0009		0.0336	
6.0	380	0.5	0.0117	0.0065	0.0070	0.0005	0.0035	0.0014	0.0028	0.0546		0.8661	0.0029		0.0430	
10.5	360	1.0	0.0029	0.0016	0.0018	0.0001	0.0006	0.0001	0.0002	0.0153		0.9401	0.0065		0.0309	

Table A-8 - Gas Analysis Halder-Iopson IK-581 Screening Test

CONDITIONS			(mol fractions)												
P (MPa)	T (°C)	LHSV (hr ⁻¹)	CH ₄	C ₂ H ₆	C ₃ H ₈	i-C ₄	n-C ₄	i-C ₅	n-C ₅	C ₆ +	CO ₂	H ₂	N ₂	O ₂	H ₂ S
10.5	360	1.0	0.0031	0.0016	0.0017	0.0001	0.0006	0.0001	0.0001	0.0029		0.9608	0.0016		0.0274
10.5	370	1.0	0.0330	0.0024	0.0026	0.0001	0.0009	0.0002	0.0003	0.0028		0.9559	0.0030		0.0285
10.5	380	1.0	0.0055	0.0038	0.0043	0.0002	0.0014	0.0004	0.0006	0.0030		0.9483	0.0027		0.0298
10.5	360	0.7	0.0028	0.0023	0.0024	0.0001	0.0007	0.0001	0.0003	0.0021		0.9534	0.0059		0.0299
10.5	370	0.7	0.0050	0.0033	0.0035	0.0002	0.0012	0.0003	0.0006	0.0035		0.9451	0.0083		0.0290
10.5	380	0.7	0.0081	0.0052	0.0057	0.0003	0.0019	0.0004	0.0006	0.0040		0.9399	0.0032		0.0307
10.5	360	0.5	0.0070	0.0055	0.0062	0.0003	0.0020	0.0005	0.0008	0.0062		0.9146			0.0569
10.5	370	0.5	0.0070	0.0046	0.0054	0.0003	0.0018	0.0004	0.0006	0.0047		0.9427	0.0023		0.0302
10.5	380	0.5													
6.0	360	1.0	0.0030	0.0014	0.0006	0.0001	0.0006	0.0002	0.0003	0.0017		0.9559	0.0063		0.0300
6.0	370	1.0	0.0029	0.0021	0.0025	0.0001	0.0008	0.0003	0.0003	0.0023		0.9523	0.0042		0.0323
6.0	380	1.0	0.0049	0.0031	0.0036	0.0002	0.0011	0.0003	0.0004	0.0033		0.9475	0.0027		0.0330
6.0	360	0.7	0.0032	0.0021	0.0025	0.0001	0.0007	0.0001	0.0003	0.0030		0.9518	0.0036		0.0327
6.0	370	0.7	0.0058	0.0030	0.0032	0.0001	0.0010	0.0002	0.0004	0.0026		0.9468	0.0043		0.0325
6.0	380	0.7	0.0078	0.0043	0.0047	0.0002	0.0015	0.0004	0.0006	0.0044		0.9321	0.0103		0.0337
6.0	360	0.5	0.0036	0.0026	0.0028	0.0001	0.0009	0.0002	0.0005	0.0031		0.9521	0.0019		0.0323
6.0	370	0.5	0.0096	0.0044	0.0049	0.0003	0.0015	0.0004	0.0006	0.0049		0.9321	0.0065		0.0347
6.0	380	0.5	0.0125	0.0061	0.0061	0.0004	0.0023	0.0005	0.0008	0.0048		0.9240	0.0092		0.0333
10.5	360	1.0													

Table A-9 - Gas Analysis Cyanamid HCN-30 Screening Test

CONDITIONS			(mol fractions)												
P (MPa)	T (°C)	LHSV (hr ⁻¹)	CH ₄	C ₂ H ₆	C ₃ H ₈	i-C ₄	n-C ₄	i-C ₅	n-C ₅	C ₆ +	CO ₂	H ₂	N ₂	O ₂	H ₂ S
10.5	360	1.0	0.0015	0.0012	0.0016	0.0001	0.0005	0.0001	0.0003	0.0020		0.9616	0.0016		0.0297
10.5	370	1.0	0.0027	0.0019	0.0023	0.0001	0.0007	0.0002	0.0003	0.0016		0.9510	0.0094		0.0298
10.5	380	1.0	0.0051	0.0030	0.0035	0.0002	0.0012	0.0003	0.0005	0.0033		0.9403	0.0122		0.0304
10.5	360	0.7	0.0023	0.0018	0.0020	0.0001	0.0006	0.0001	0.0002	0.0019		0.9583	0.0020		0.0307
10.5	370	0.7	0.0037	0.0025	0.0031	0.0001	0.0010	0.0002	0.0004	0.0012		0.9533	0.0027		0.0319
10.5	380	0.7	0.0063	0.0039	0.0036	0.0002	0.0016	0.0003	0.0005	0.0029		0.9366	0.0122		0.0318
10.5	360	0.5	0.0035	0.0024	0.0026	0.0001	0.0010	0.0001	0.0008	0.0023		0.9551	0.0024		0.0298
10.5	370	0.5	0.0061	0.0036	0.0042	0.0002	0.0014	0.0004	0.0006	0.0031		0.9376	0.0114		0.0315
10.5	380	0.5	0.0111	0.0061	0.0063	0.0004	0.0022	0.0005	0.0007	0.0038		0.9326	0.0032		0.0330
6.0	360	1.0	0.0013	0.0013	0.0015	0.0001	0.0005	0.0001	0.0002	0.0029		0.9567	0.0032		0.0322
6.0	370	1.0	0.0019	0.0019	0.0021	0.0001	0.0008	0.0002	0.0004	0.0020		0.9456	0.0117		0.0322
6.0	380	1.0	0.0027	0.0027	0.0005	0.0001	0.0010	0.0002	0.0005	0.0039		0.9438	0.0081		0.0310
6.0	360	0.7	0.0016	0.0016	0.0015	0.0001	0.0006	0.0001	0.0003	0.0033		0.9590	0.0017		0.0300
6.0	370	0.7	0.0024	0.0024	0.0030	0.0001	0.0010	0.0003	0.0004	0.0031		0.9493	0.0050		0.0310
6.0	380	0.7	0.0038	0.0038	0.0014	0.0002	0.0015	0.0004	0.0007	0.0043		0.9373	0.0094		0.0327
6.0	360	0.5	0.0033	0.0022	0.0018	0.0001	0.0009	0.0003	0.0003	0.0032		0.9544	0.0019		0.0317
6.0	370	0.5	0.0065	0.0037	0.0043	0.0002	0.0014	0.0003	0.0008	0.0036		0.9327	0.0139		0.0327
6.0	380	0.5	0.0110	0.0057	0.0045	0.0004	0.0024	0.0005	0.0009	0.0066		0.9126	0.0153		0.0360
10.5	360	1.0	0.0017	0.0013	0.0013	0.0001	0.0005	0.0001	0.0003	0.0031		0.9600	0.0029		0.0287

CATALYST: CYANAMID HDS-20							
CONDITIONS			Actual LHSV (hr ⁻¹)	Liquid Yield (wt % FF)	Gas Yield (wt % FF)	H ₂ Csump (gmol/g)	Duration (hr)
P (MPa)	T (°C)	LHSV (hr ⁻¹)					
10.5	360	1.0	0.92	97.8	2.5	0.0278	4.33
10.5	370	1.0	0.83	99.3	4.7	0.0148	6.00
10.5	380	1.0	1.00	94.2	5.1	0.0123	6.00
10.5	360	0.75	0.84	89.9	3.5	0.0048	7.00
10.5	370	0.75	0.72	100.0	5.0	0.0119	6.00
10.5	380	0.75	0.73	96.8	5.4	0.0119	6.00
10.5	360	0.5	0.55	95.2	5.0	0.0160	6.00
10.5	370	0.5	0.49	96.3	5.3	0.0173	6.00
10.5	380	0.5	0.51	98.1	5.4	0.0167	6.00
6.0	360	1.0	0.90	98.0	4.8	0.0093	6.00
6.0	370	1.0	0.94	96.2	5.7	0.0099	6.00
6.0	380	1.0	0.96	92.5	5.8	0.0100	6.00
6.0	360	0.75	0.77	93.5	5.3	0.0094	6.00
6.0	370	0.75	0.74	97.6	9.7	0.0112	6.00
6.0	380	0.75	0.75	94.3	7.9	0.0106	6.00
6.0	360	0.5	0.53	94.6	4.8	0.0118	6.00
6.0	370	0.5	0.51	98.6	6.5	0.0112	6.00
6.0	380	0.5	0.50	96.1	7.6	0.0111	6.00
10.5	360	1.0	1.04	95.0	4.4	0.0109	16.88
Total Time (hr) = 124.21							

Total Time (hr) = 124.21

Table A-11 - Material Balances Ketjen KF-840
Screening Test

CATALYST: KETJEN KF-840

[illegible]

Table A-12 - Material Balances Halder-Topsoe TK-581 Screening Test							
CATALYST: HALDER-TOPSOE TK-581							
CONDITIONS			Actual LHSV hr⁻¹	Liquid Yield (wt % FF)	Gas Yield (wt % FF)	H₂ Csump (gmol/g)	Duration (hr)
P (MPa)	T (°C)	LHSV (hr⁻¹)					
10.5	360	1.0	0.97	96.8	4.4	0.0115	5.45
10.5	370	1.0	0.96	97.2	4.9	0.0124	10.45
10.5	380	1.0	1.01	95.7	5.3	0.0127	6.00
10.5	360	0.75	0.76	95.6	5.1	0.0099	6.00
10.5	370	0.75	0.71	96.4	5.9	0.0142	11.17
10.5	380	0.75	0.78	94.1	5.7	0.0140	6.00
10.5	360	0.5	0.55	95.1	7.7	0.0145	6.58
10.5	370	0.5	0.50	95.9	5.6	0.0158	10.42
10.5	380	0.5	0.50	99.1			6.00
6.0	360	1.0	1.03	96.1	4.6	0.0102	6.00
6.0	370	1.0	1.02	96.2	5.2	0.0104	6.00
6.0	380	1.0	1.01	96.0	5.8	0.0111	11.00
6.0	360	0.75	0.79	90.8	3.9	0.0110	6.00
6.0	370	0.75	0.75	95.4	5.6	0.0116	6.00
6.0	380	0.75	0.74	95.3	7.3	0.0122	9.83
6.0	360	0.5	0.55	95.0	5.1	0.0091	6.00
6.0	370	0.5	0.52	95.1	7.6	0.0099	6.33
6.0	380	0.5	0.51	94.2	8.3	0.0104	10.45
10.5	360	1.0	1.00	97.4	4.7	0.0105	6.00
Total Time (hr) = 141.68							

Total Time (hr) = 141.68

CATALYST: CYANAMID HDN-30							
CONDITIONS			Actual LHSV hr ⁻¹	Liquid Yield (wt % FF)	Gas Yield (wt % FF)	H ₂ Csump (gmol/g)	Duration (hr)
P (MPa)	T (°C)	LHSV (hr ⁻¹)					
10.5	360	1.0	0.98	96.1	4.3	0.0112	6.00
10.5	370	1.0	0.99	95.7	5.0	0.0118	6.00
10.5	380	1.0	0.98	96.2	6.3	0.0127	11.00
10.5	360	0.75	0.74	95.7	4.4	0.0123	6.30
10.5	370	0.75	0.80	93.7	4.3	0.0121	6.00
10.5	380	0.75	0.76	96.1	6.1	0.0141	10.45
10.5	360	0.5	0.51	96.8	4.2	0.0144	6.00
10.5	370	0.5	0.50	96.6	5.7	0.0156	6.58
10.5	380	0.5	0.51	95.6	6.0	0.0159	11.00
6.0	360	1.0	1.01	97.6	5.4	0.0073	6.00
6.0	370	1.0	1.01	96.3	6.3	0.0079	6.00
6.0	380	1.0	1.01	95.4	6.5	0.0081	11.17
6.0	360	0.75	0.76	95.2	5.0	0.0082	6.00
6.0	370	0.75	0.75	95.6	6.0	0.0089	6.00
6.0	380	0.75	0.79	95.0	6.8	0.0090	11.00
6.0	360	0.5	0.56	97.2	4.9	0.0082	6.00
6.0	370	0.5	0.52	95.8	7.4	0.0090	6.42
6.0	380	0.5	0.53	94.0	9.3	0.0093	10.83
10.5	360	1.0	0.99	96.1	5.5	0.0098	6.00

Total Time (hr) = 144.75

CATALYST: CYANAMID HDN-30							
CONDITIONS			Actual LHSV hr ⁻¹	Liquid Yield (wt % FF)	Gas Yield (wt % FF)	H ₂ Csump (gmol/g)	Duration (hr)
P (MPa)	T (°C)	LHSV (hr ⁻¹)					
10.5	360	1.0	0.98	96.1	4.3	0.0112	6.00
10.5	370	1.0	0.99	95.7	5.0	0.0118	6.00
10.5	380	1.0	0.98	96.2	6.3	0.0127	11.00
10.5	360	0.75	0.74	95.7	4.4	0.0123	6.30
10.5	370	0.75	0.80	93.7	4.3	0.0121	6.00
10.5	380	0.75	0.76	96.1	6.1	0.0141	10.45
10.5	360	0.5	0.51	96.8	4.2	0.0144	6.00
10.5	370	0.5	0.50	96.6	5.7	0.0156	6.58
10.5	380	0.5	0.51	95.6	6.0	0.0159	11.00
6.0	360	1.0	1.01	97.6	5.4	0.0073	6.00
6.0	370	1.0	1.01	96.3	6.3	0.0079	6.00
6.0	380	1.0	1.01	95.4	6.5	0.0081	11.17
6.0	360	0.75	0.76	95.2	5.0	0.0082	6.00
6.0	370	0.75	0.75	95.6	6.0	0.0089	6.00
6.0	380	0.75	0.79	95.0	6.8	0.0090	11.00
6.0	360	0.5	0.56	97.2	4.9	0.0082	6.00
6.0	370	0.5	0.52	95.8	7.4	0.0090	6.42
6.0	380	0.5	0.53	94.0	9.3	0.0093	10.83
10.5	360	1.0	0.99	96.1	5.5	0.0098	6.00

Total Time (hr) = 144.75

CATALYST: CYANAMID HDN-30							
CONDITIONS			Actual LHSV hr ⁻¹	Liquid Yield (wt % FF)	Gas Yield (wt % FF)	H ₂ Csump (gmol/g)	Duration (hr)
P (MPa)	T (°C)	LHSV (hr ⁻¹)					
10.5	360	1.0	0.98	96.1	4.3	0.0112	6.00
10.5	370	1.0	0.99	95.7	5.0	0.0118	6.00
10.5	380	1.0	0.98	96.2	6.3	0.0127	11.00
10.5	360	0.75	0.74	95.7	4.4	0.0123	6.30
10.5	370	0.75	0.80	93.7	4.3	0.0121	6.00
10.5	380	0.75	0.76	96.1	6.1	0.0141	10.45
10.5	360	0.5	0.51	96.8	4.2	0.0144	6.00
10.5	370	0.5	0.50	96.6	5.7	0.0156	6.58
10.5	380	0.5	0.51	95.6	6.0	0.0159	11.00
6.0	360	1.0	1.01	97.6	5.4	0.0073	6.00
6.0	370	1.0	1.01	96.3	6.3	0.0079	6.00
6.0	380	1.0	1.01	95.4	6.5	0.0081	11.17
6.0	360	0.75	0.76	95.2	5.0	0.0082	6.00
6.0	370	0.75	0.75	95.6	6.0	0.0089	6.00
6.0	380	0.75	0.79	95.0	6.8	0.0090	11.00
6.0	360	0.5	0.56	97.2	4.9	0.0082	6.00
6.0	370	0.5	0.52	95.8	7.4	0.0090	6.42
6.0	380	0.5	0.53	94.0	9.3	0.0093	10.83
10.5	360	1.0	0.99	96.1	5.5	0.0098	6.00

Total Time (hr) = 144.75

**Table A-14 - Day One - Liquid Products Shell S-424
Catalyst Long-Term Test**

Temp (°C)	CONDITIONS LHSV (hr ⁻¹)	Press (MPa)	Period (#)	Density (kg/m ³)	Sulphur (wt %)	Nitrogen (wppm)
375	0.75	8.75	F86-07B	861.2	0.08	4.9
360	1.0	10.5	F86-07C	886.7	0.03	76.4
390	1.0	10.5	F86-07D	873.6	0.01	26.7
360	0.5	10.5	F86-07E	897.6	0.10	123.0
390	0.5	10.5	F86-07F	878.2	0.03	7.0
375	0.75	8.75	F86-07G	902.8	0.18	400.0
360	1.0	6.0	F86-07H	915.6	0.49	1117.0
390	1.0	6.0	F86-07I	903.7	0.08	509.0
360	0.5	6.0	F86-07J	909.6	0.28	772.0
390	0.5	6.0	F86-07K	890.7	0.07	101.0
375	0.75	8.75	F86-07L	901.9	0.20	317.0

**Table A-15 - Day Two - Liquid Products Shell S-424
Catalyst Long-Term Test**

Temp (°C)	CONDITIONS LHSV (hr ⁻¹)	Press (MPa)	Period (#)	Density (kg/m ³)	Sulphur (wt %)	Nitrogen (wppm)
375	0.75	8.75	F86-07B	865.1	0.04	4.6
360	1.0	10.5	F86-07C	885.8	0.04	78.9
390	1.0	10.5	F86-07D	880.0	<0.01	40.5
360	0.5	10.5	F86-07E	900.1	0.16	201.0
390	0.5	10.5	F86-07F	874.6	0.02	4.1
375	0.75	8.75	F86-07G			
360	1.0	6.0	F86-07H	915.5	0.46	1232.0
390	1.0	6.0	F86-07I	904.2	0.13	547.0
360	0.5	6.0	F86-07J	908.8	0.22	708.0
390	0.5	6.0	F86-07K	890.6	0.04	67.2
375	0.75	8.75	F86-07L			

**Table A-16 - Day Three - Liquid Products Shell S-424
Catalysts Long-Term Test**

Temp (°C)	CONDITIONS LHSV (hr ⁻¹)	Press (MPa)	Period (#)	Density (kg/m ³)	Sulphur (wt %)	Nitrogen (wppm)
375	0.75	8.75	F86-07B			
360	1.0	10.5	F86-07C	887.2	0.04	75.2
390	1.0	10.5	F86-07D	878.3	0.01	29.4
360	0.5	10.5	F86-07E	902.3	0.18	283.0
390	0.5	10.5	F86-07F	875.2	0.02	8.9
375	0.75	8.75	F86-07G			
360	1.0	6.0	F86-07H	914.8	0.44	1193.0
390	1.0	6.0	F86-07I	902.8	0.08	461.0
360	0.5	6.0	F86-07J	908.6	0.21	681.0
390	0.5	6.0	F86-07K	885.5	0.06	33.9
375	0.75	8.75	F86-07L			

Table A-17 - Day One - Material Balance Shell S424
Catalysts Long-Term Test

CONDITIONS			Period #)	Actual LHSV	Liquid Prod (wt %FF)	Gas Prod (wt %FF)	Time (hr)	H ₂ Csump (gmol/g)
Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)						
375	0.75	8.75	F86-07B	0.78	91.0	6.5	24.0	0.0140
360	1.0	10.5	F86-07C	0.97	93.8	4.6	27.2	0.0118
390	1.0	10.5	F86-07D	1.04	91.2	3.0	20.4	0.0200
360	0.5	10.5	F86-07E	0.52	95.5	3.2	24.0	0.0111
390	0.5	10.5	F86-07F	0.51	96.4	3.7	24.0	0.0141
375	0.75	8.75	F86-07G	0.68	94.1	3.3	24.1	0.0222
360	1.0	6.0	F86-07H	1.04	96.5	3.7	24.0	0.0078
390	1.0	6.0	F86-07I	0.98	94.9	4.4	24.0	0.0086
360	0.5	6.0	F86-07J	0.55	94.0	4.2	24.1	0.0080
390	0.5	6.0	F86-07K	0.50	95.4	5.2	24.2	0.0107
375	0.75	8.75	F86-07L	0.76	96.4	3.3	24.0	0.0237

Table A-18 - Day Two - Material Balance Shell S-424
Catalysts Long-Term Test

CONDITIONS			Period (#)	Actual LHSV	Liquid Prod (wt %FF)	Gas Prod (wt %FF)	Time (hr)	H ₂ Csum (gmol/g)
Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)						
375	0.75	8.75	F86-07B	0.75	101.0	6.7	24.4	0.0135
360	1.0	10.5	F86-07C	0.98	96.0	4.2	19.9	0.0126
390	1.0	10.5	F86-07D	1.01	90.3	4.8	24.0	0.0120
360	0.5	10.5	F86-07E	0.49	98.5	3.4	25.1	0.0111
390	0.5	10.5	F86-07F	0.49	96.6	4.0	24.0	0.0135
375	0.75	8.75	F86-07G					
360	1.0	6.0	F86-07H	1.01	96.7	4.5	23.5	0.0024
390	1.0	6.0	F86-07I	0.98	96.8	4.0	24.0	0.0076
360	0.5	6.0	F86-07J	0.50	97.0	4.3	24.0	0.0076
390	0.5	6.0	F86-07K	0.50	93.6	5.1	23.8	0.0103
375	0.75	8.75	F86-07L					

Table A-19 - Day Three - Material Balance Shell S-424
Catalysts Long-Term Test

CONDITIONS			Period (#)	Actual LHSV	Liquid Prod (wt %FF)	Gas Prod (wt %FF)	Time (hr)	H ₂ Csump (gmol/g)
Temp (°C)	LHSV (hr ⁻¹)	Press (MPa)						
375	0.75	8.75	F86-07B					
360	1.0	10.5	F86-07C	1.00	93.9	3.9	24.7	0.0125
390	1.0	10.5	F86-07D	1.08	90.3	4.8	25.1	0.0100
360	0.5	10.5	F86-07E	0.50	96.3	3.3	24.5	00.0108
390	0.5	10.5	F86-07F	0.50	94.5	4.0	24.0	0.0128
375	0.75	8.75	F86-07G					
360	1.0	6.0	F86-07H	0.95	97.0	3.5	24.6	0.0133
390	1.0	6.0	F86-07I	1.01	96.0	4.0	24.0	0.0109
360	0.5	6.0	F86-07J	0.49	97.0	4.5	24.0	0.0077
390	0.5	6.0	F86-07K	0.50	95.1	4.9	24.0	0.0113
375	0.75	8.75	F86-07L					

APPENDIX B

FIGURES

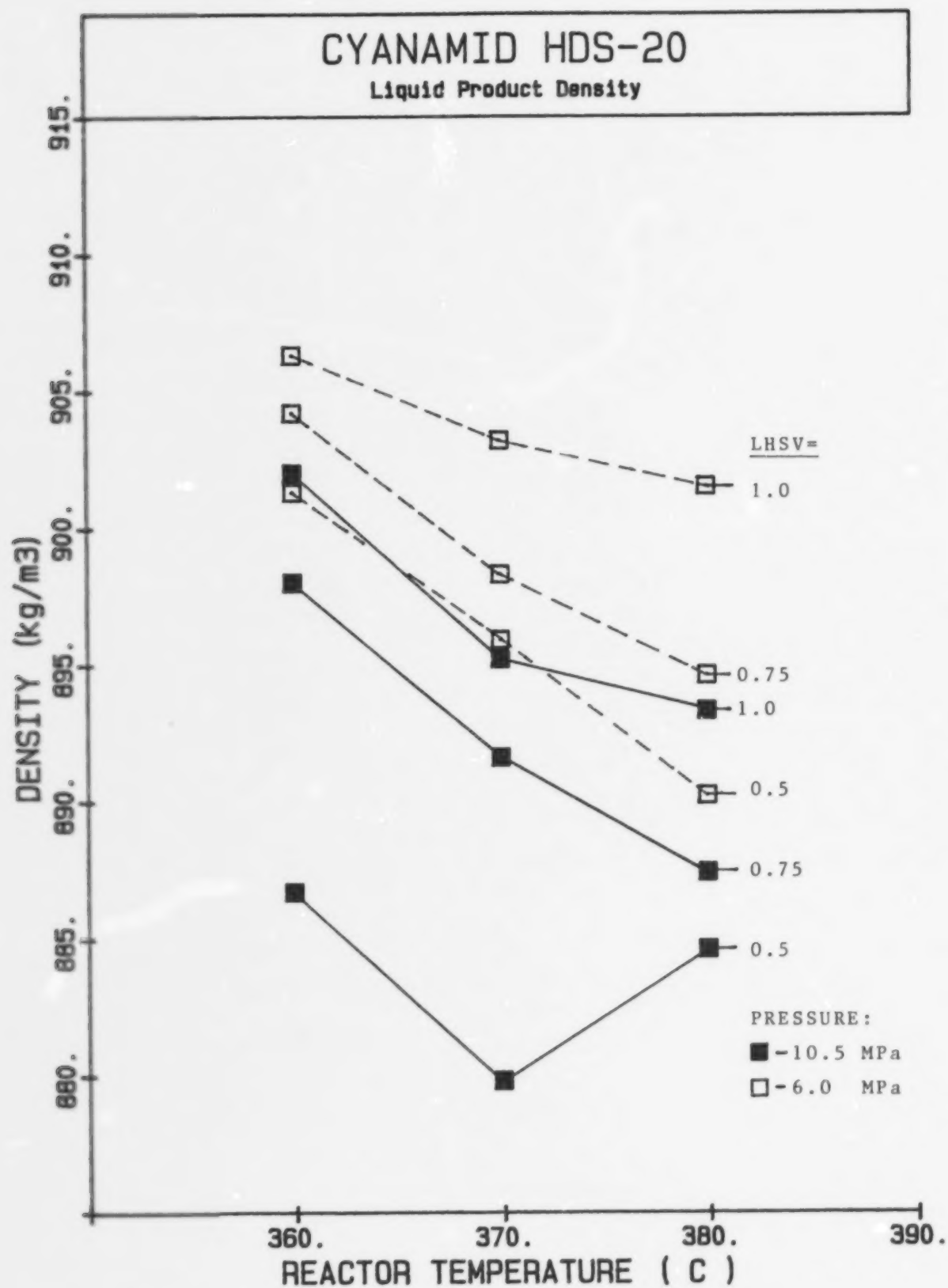


Figure B-1 - Liquid Product Density - Cyanamid HDS-20

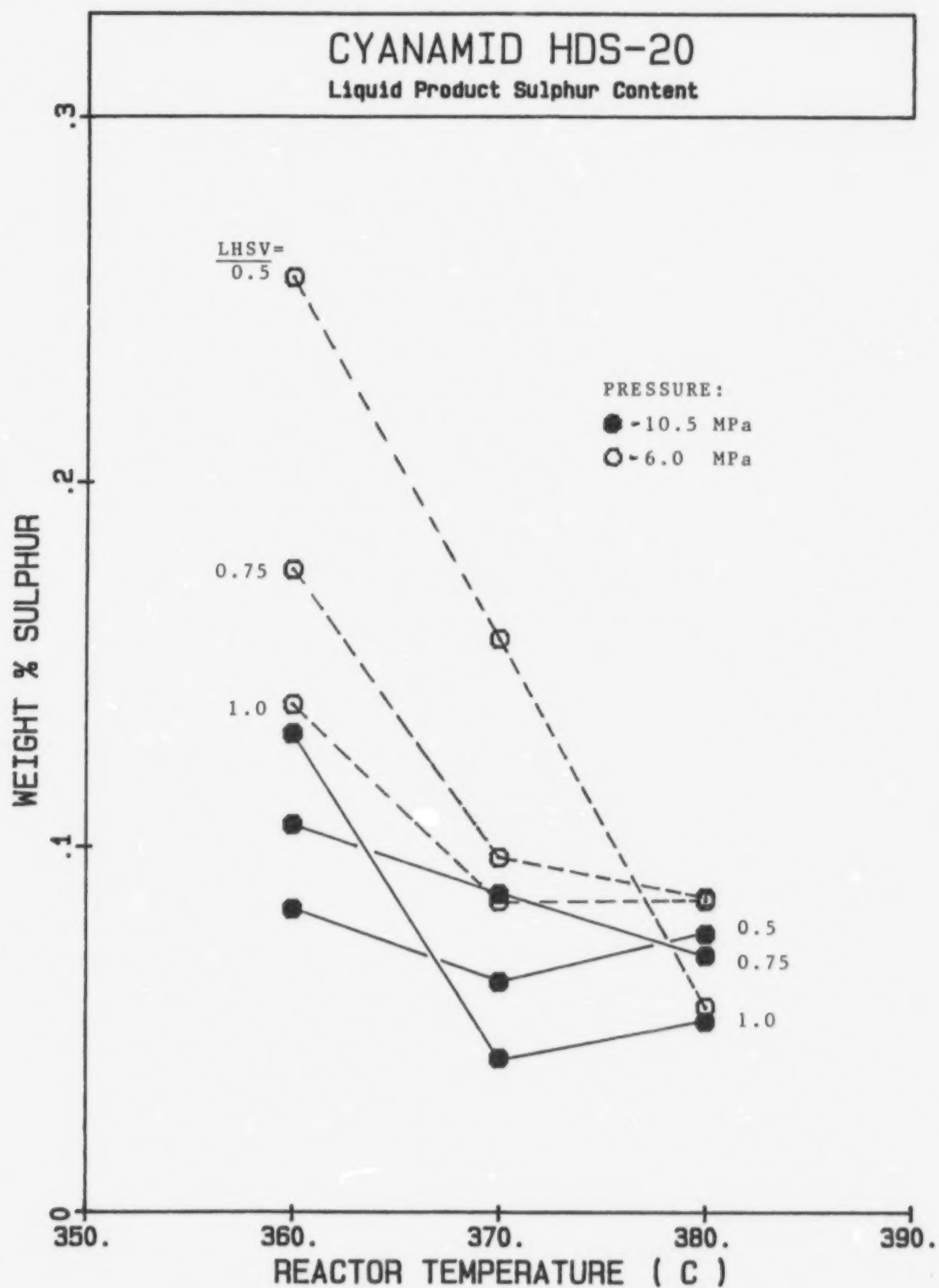


Figure B-2 - Liquid Product Sulphur Content - HDS-20

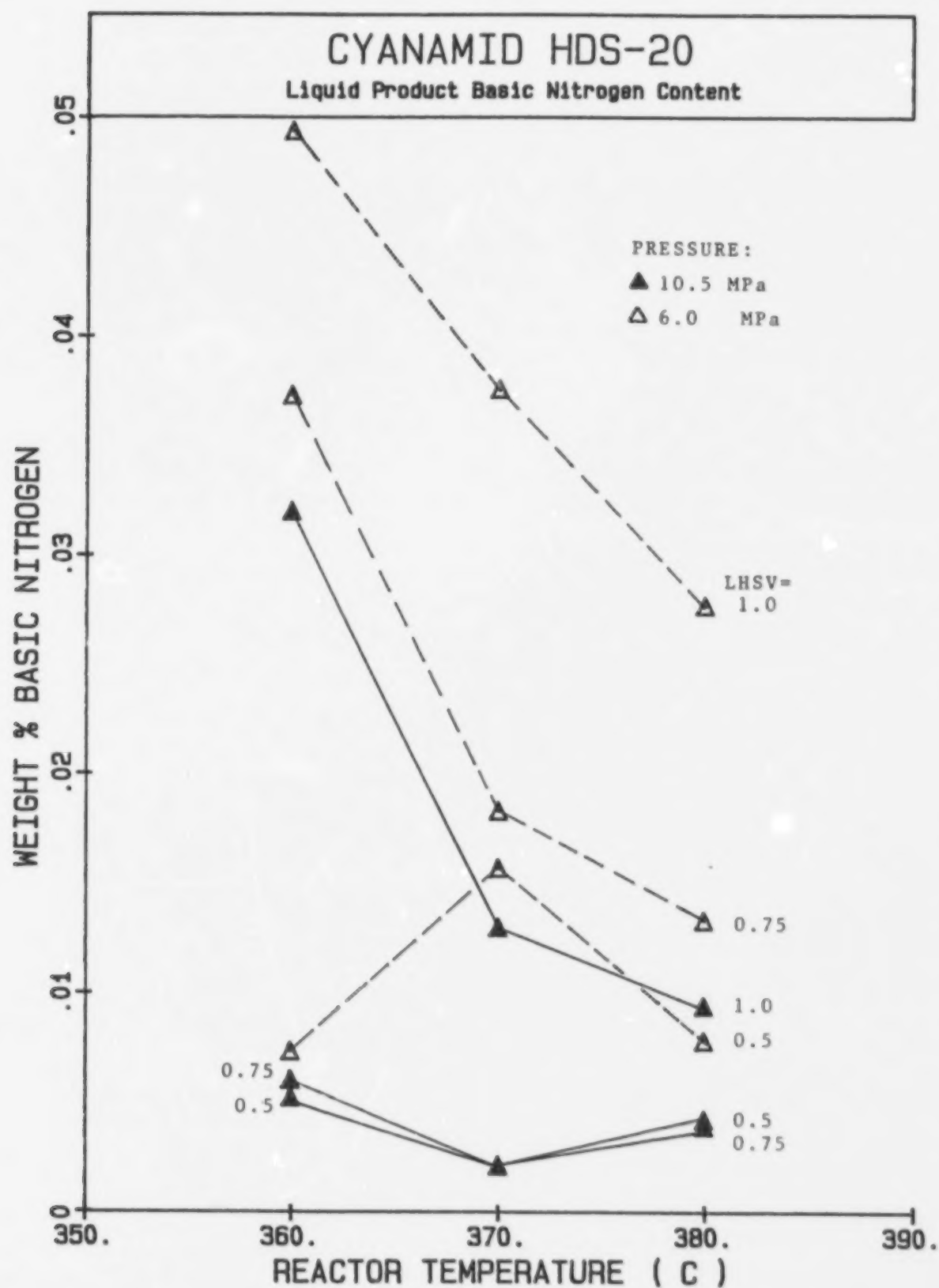


Figure B-3 - Liquid Product Nitrogen Content - HDS-20

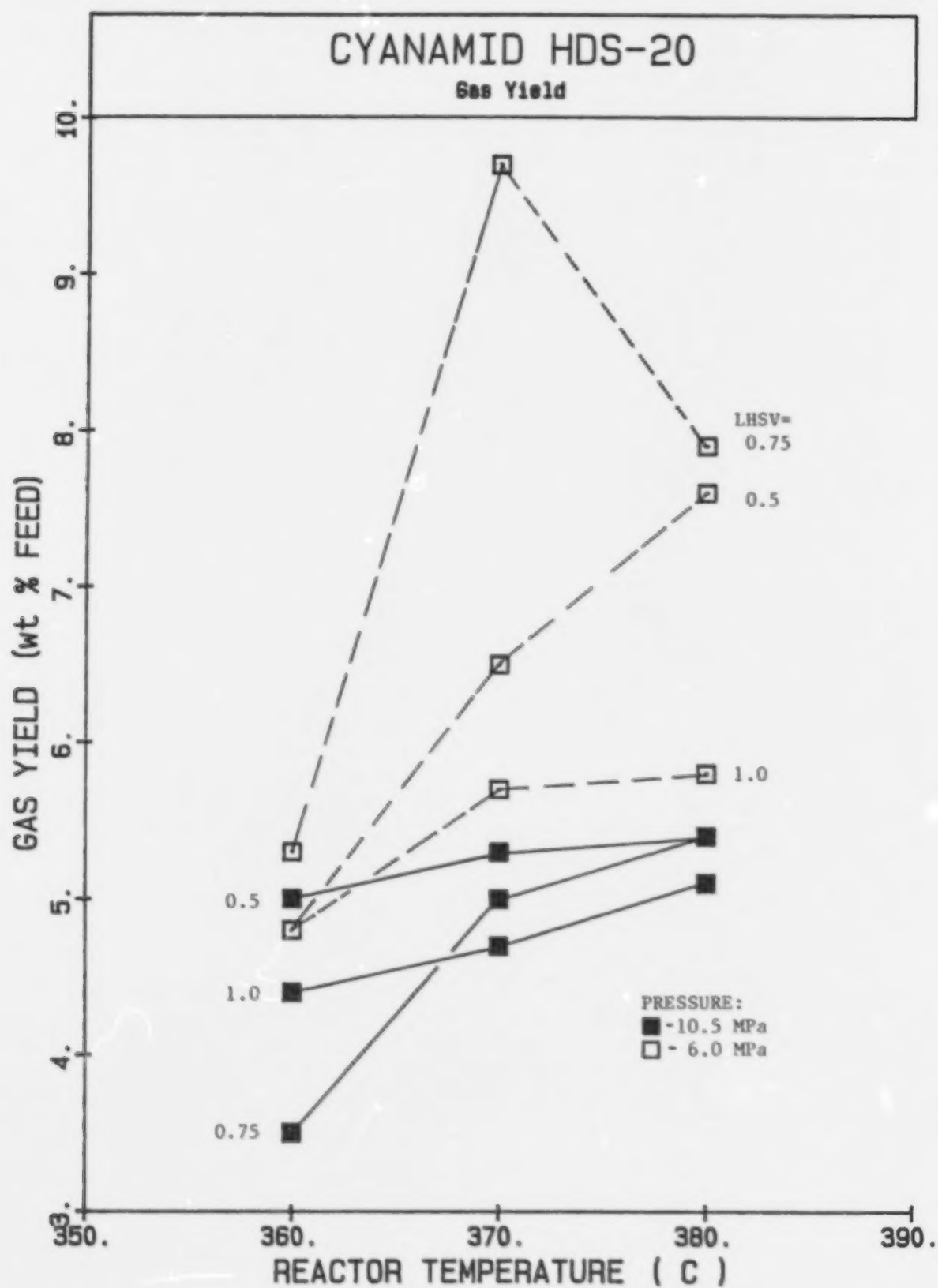


Figure B-4 - Gas Yield - HDS-20

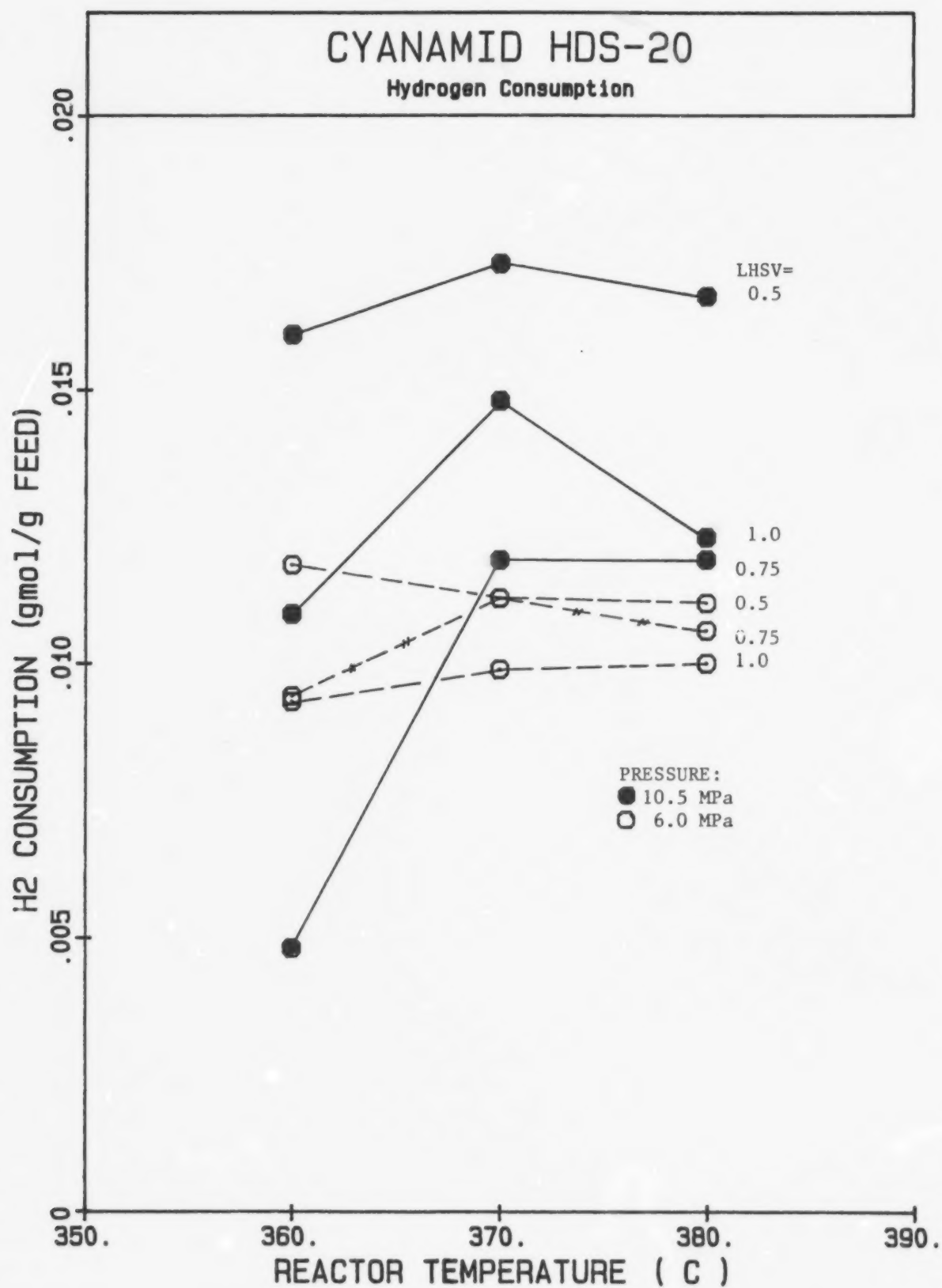


Figure B-5 - Hydrogen Consumption - HDS-20

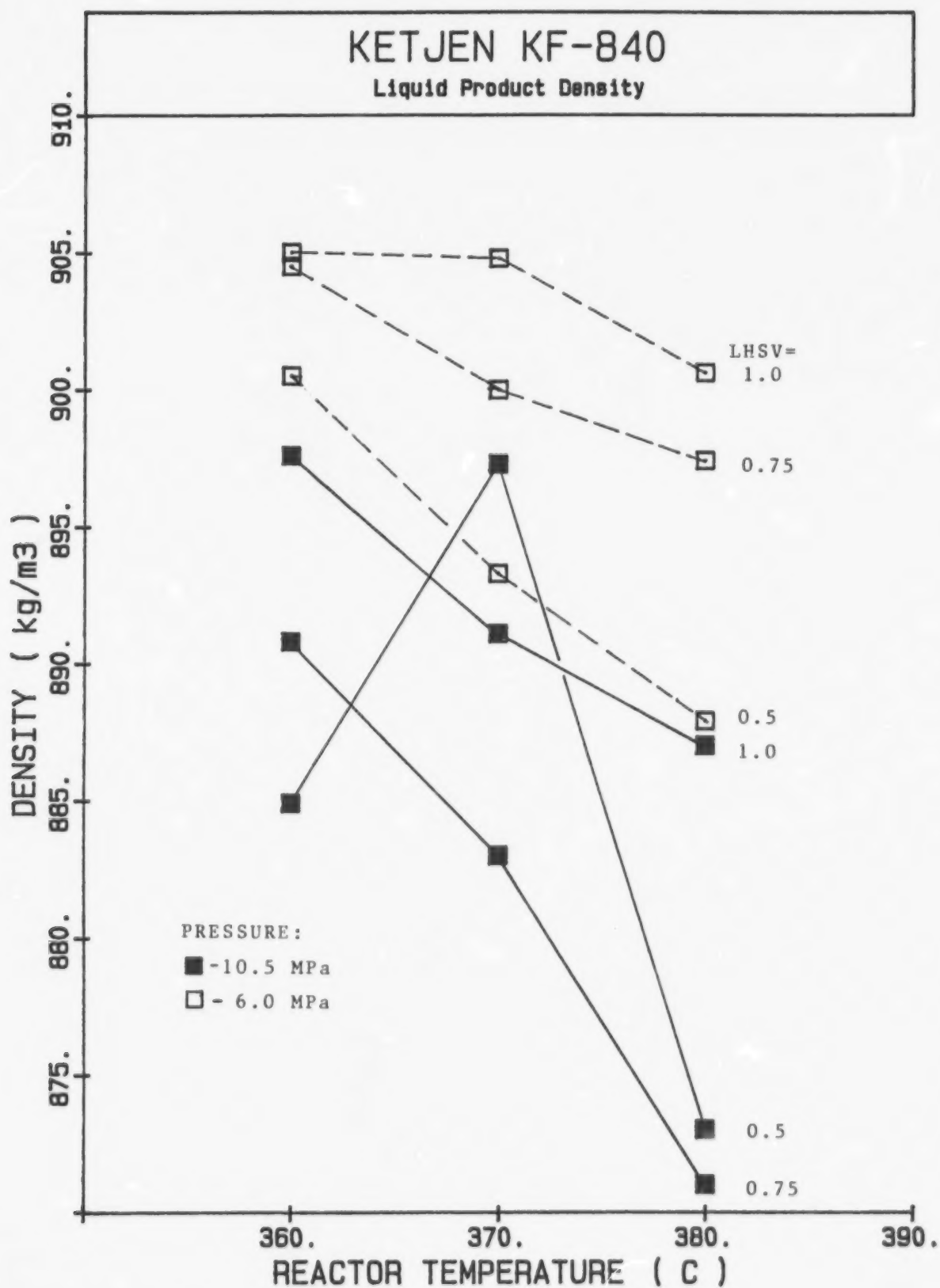


Figure B-6 - Liquid Product Density - Ketjen KF-840

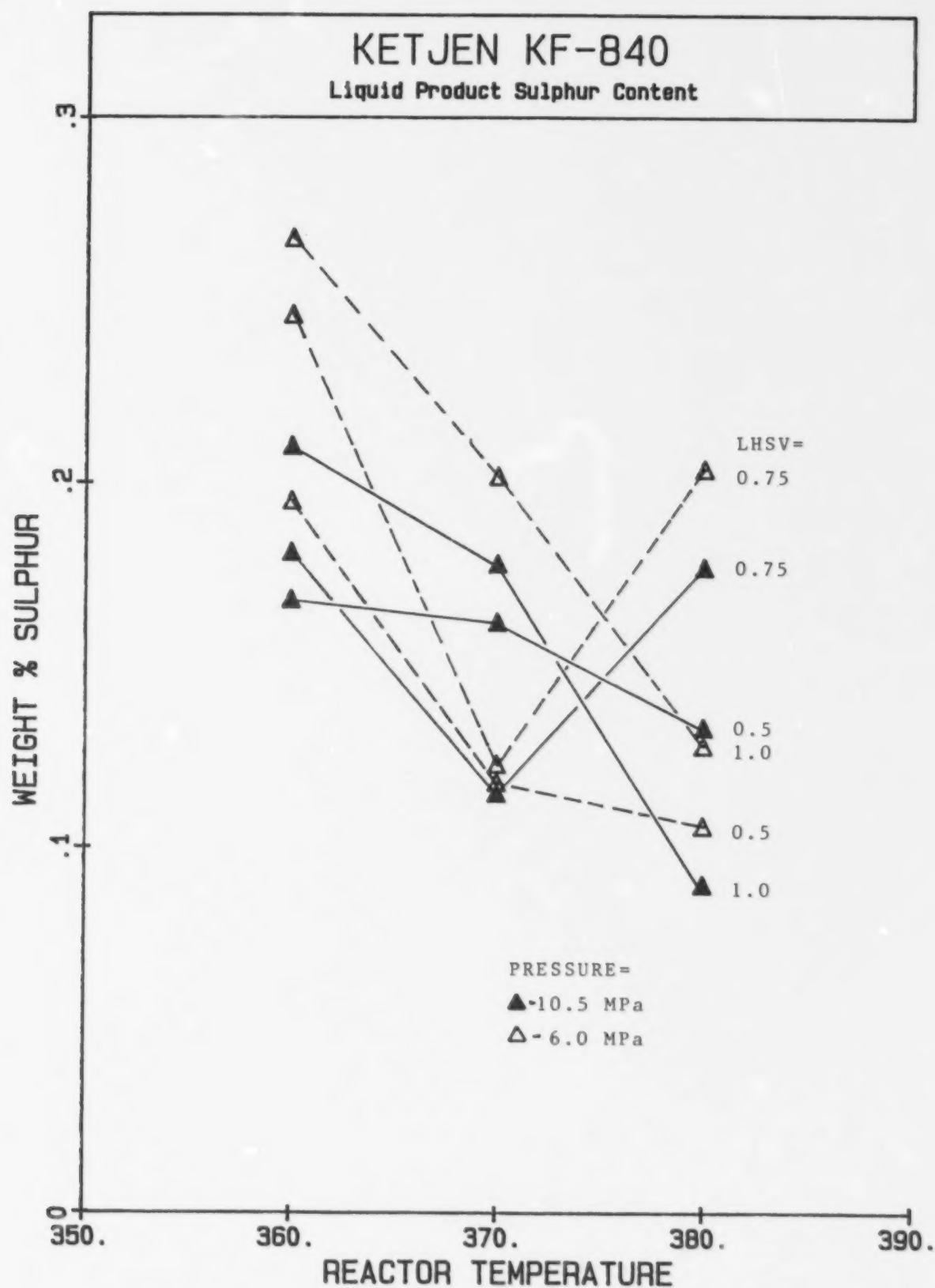


Figure B-7 - Liquid Product Sulphur Content - KF-840

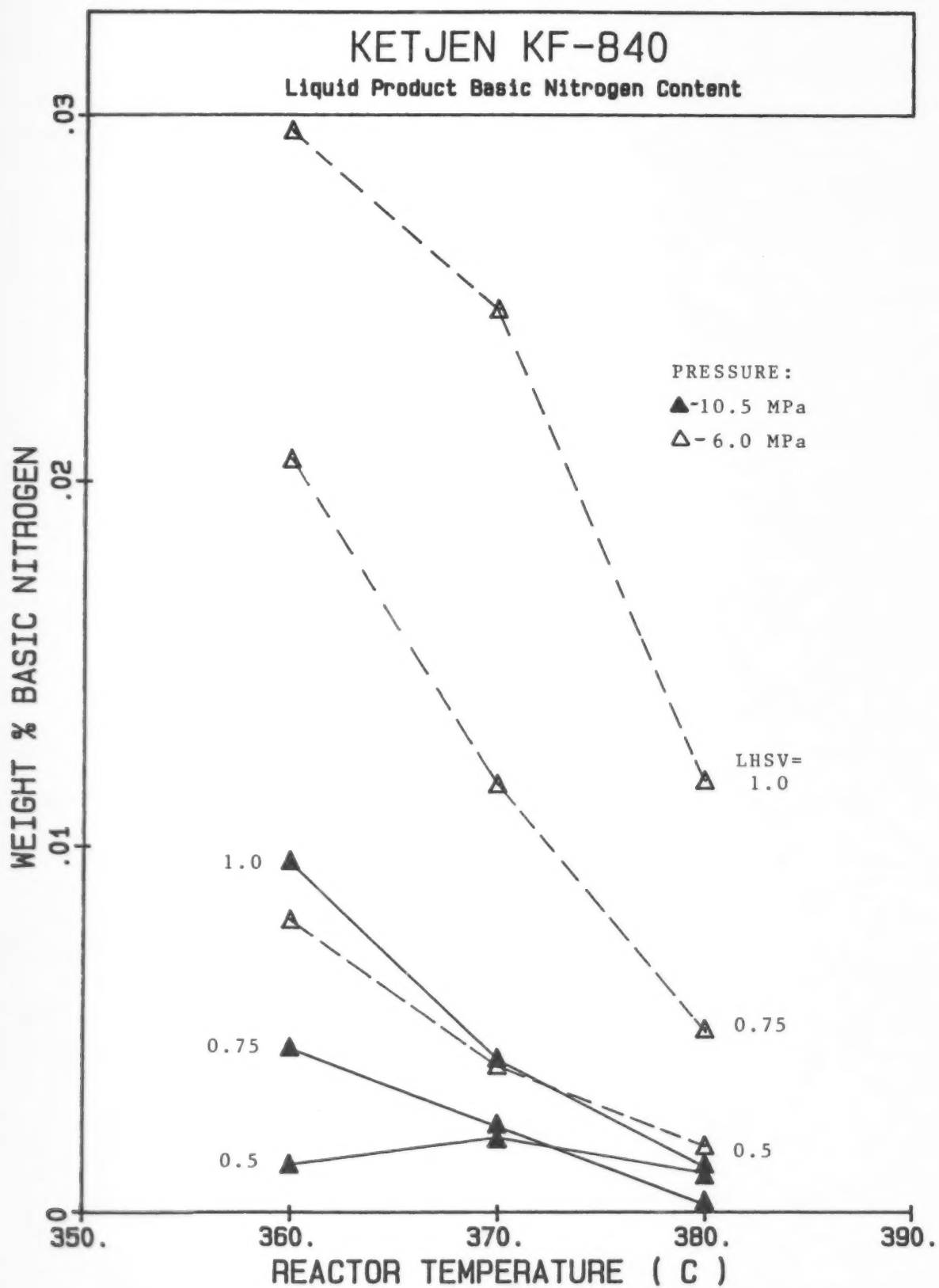


Figure B-8 - Liquid Product Basic Nitrogen Content KF-840

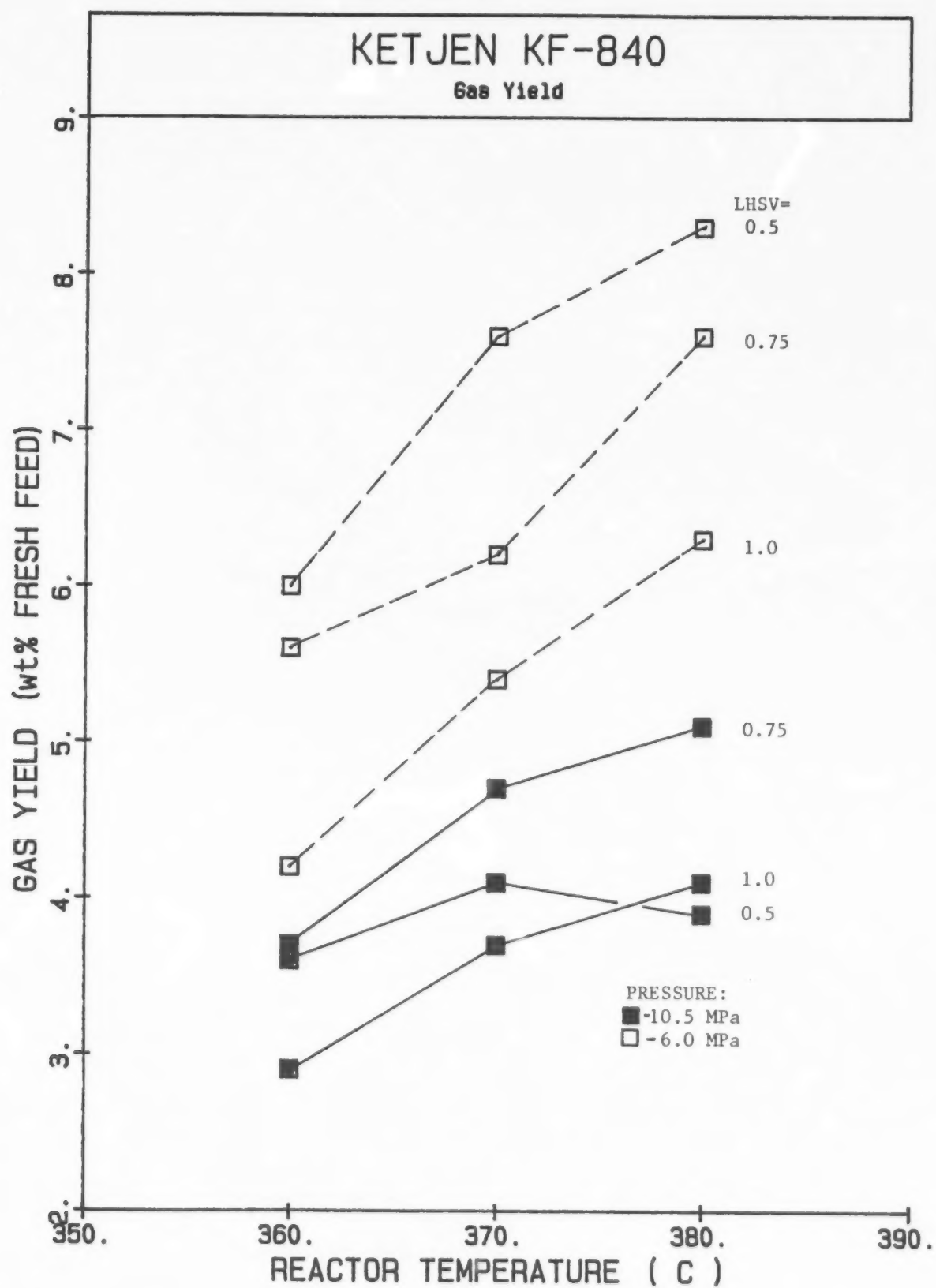


Figure B-9 - Gas Yield - KF-840

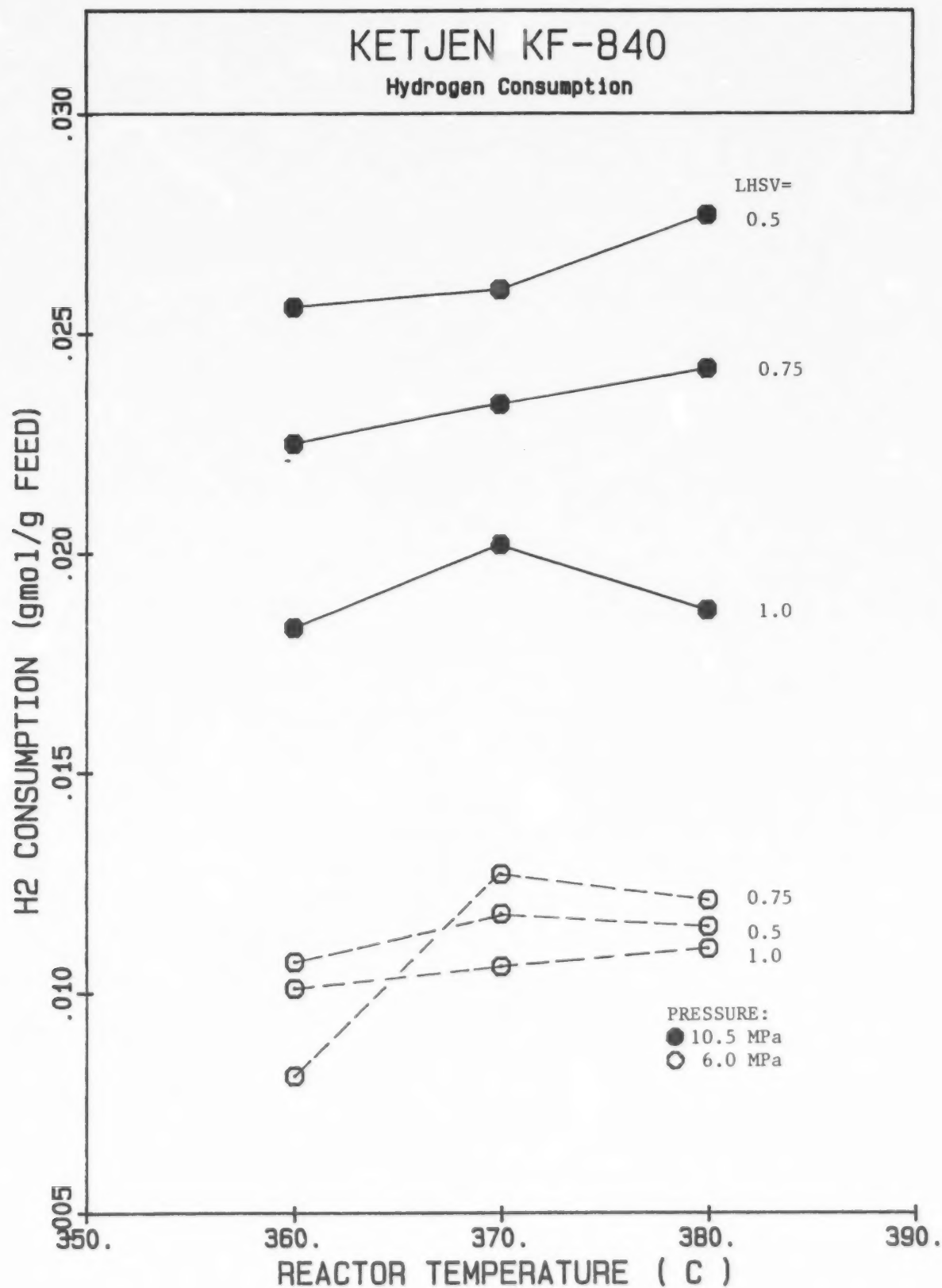


Figure B-10 - Hydrogen Consumption - KF-840

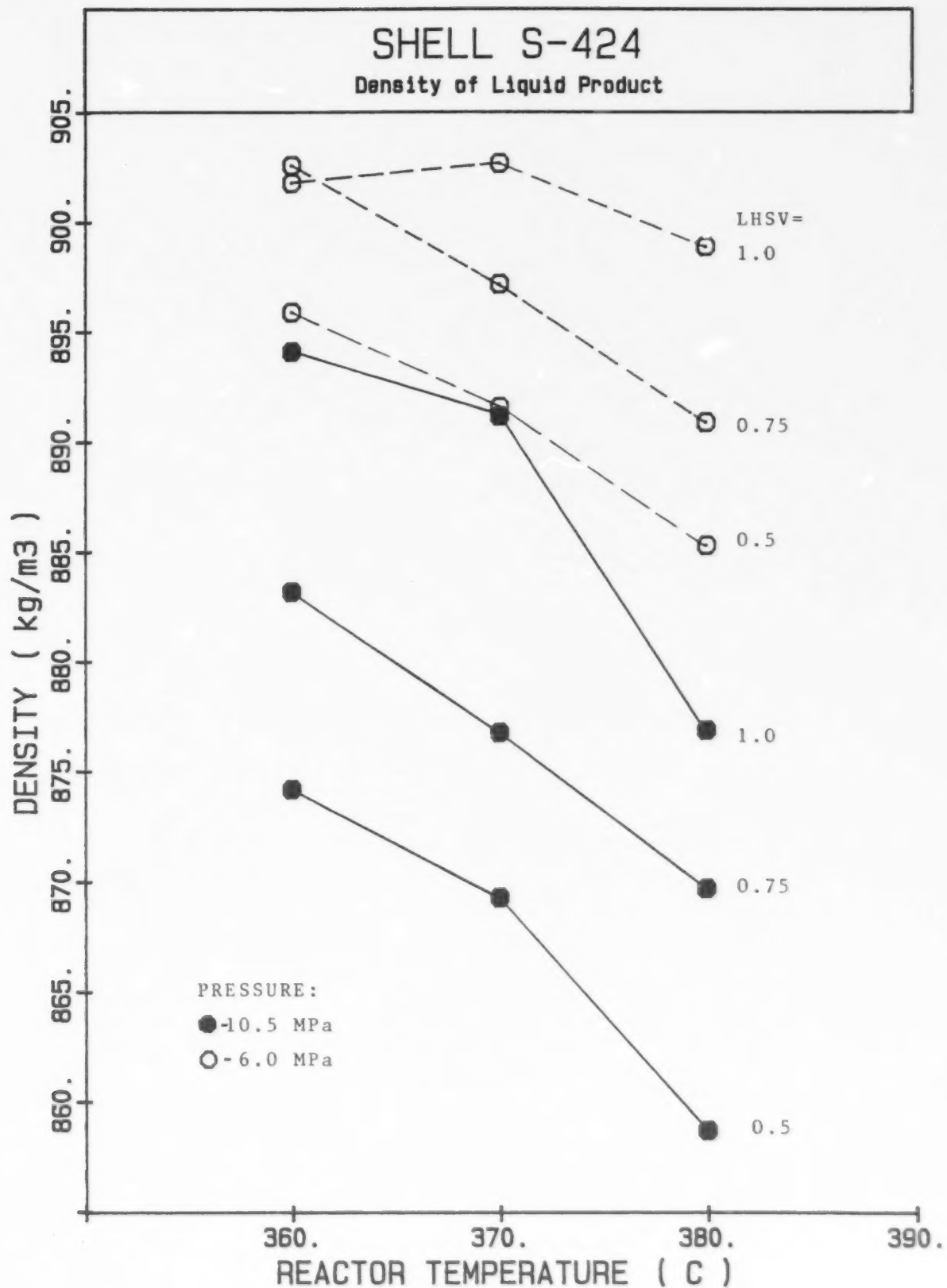


Figure B-11 - Liquid Product Density - Shell S-424

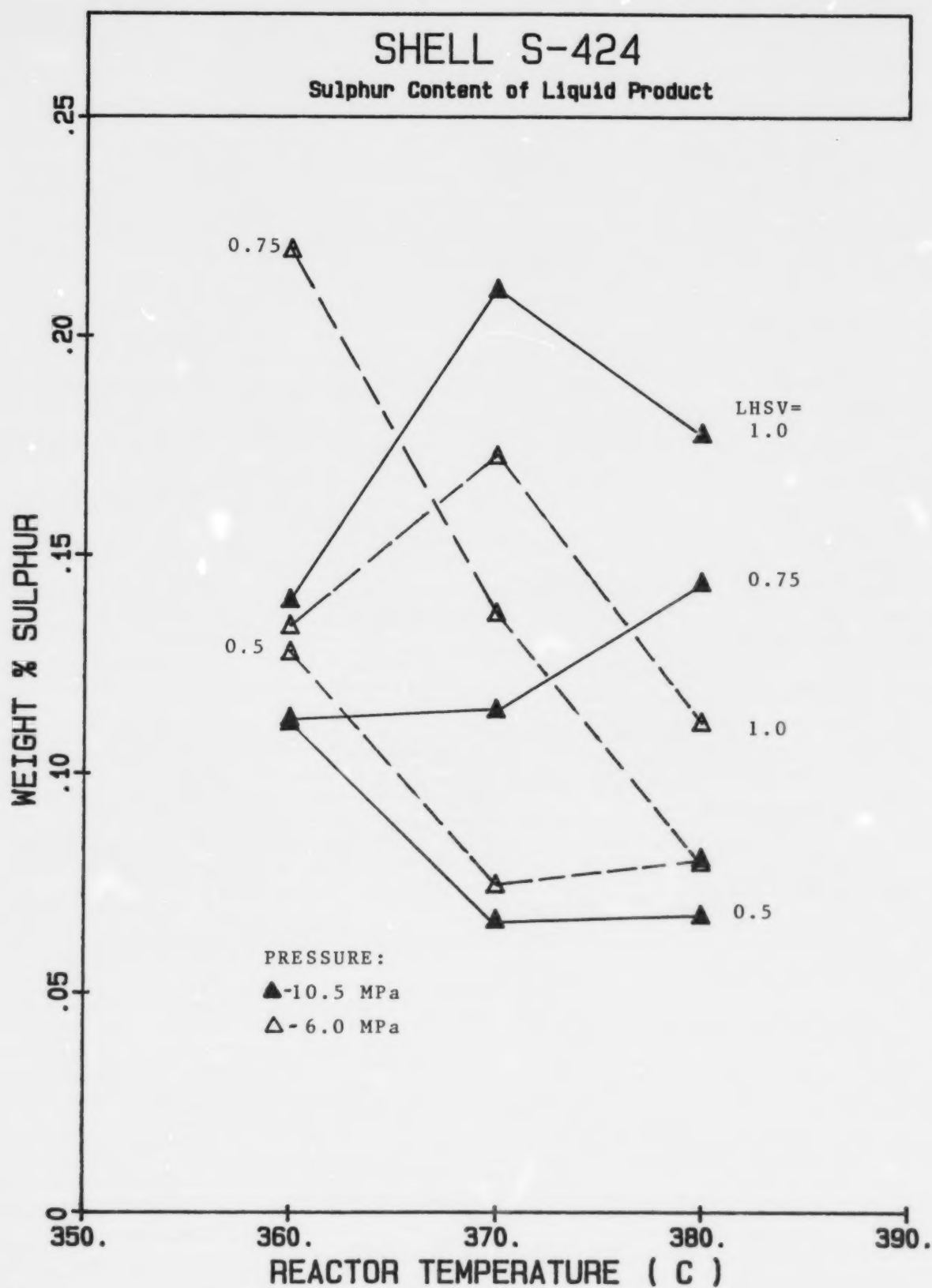


Figure B-12 - Liquid Product Sulphur Content - S-424

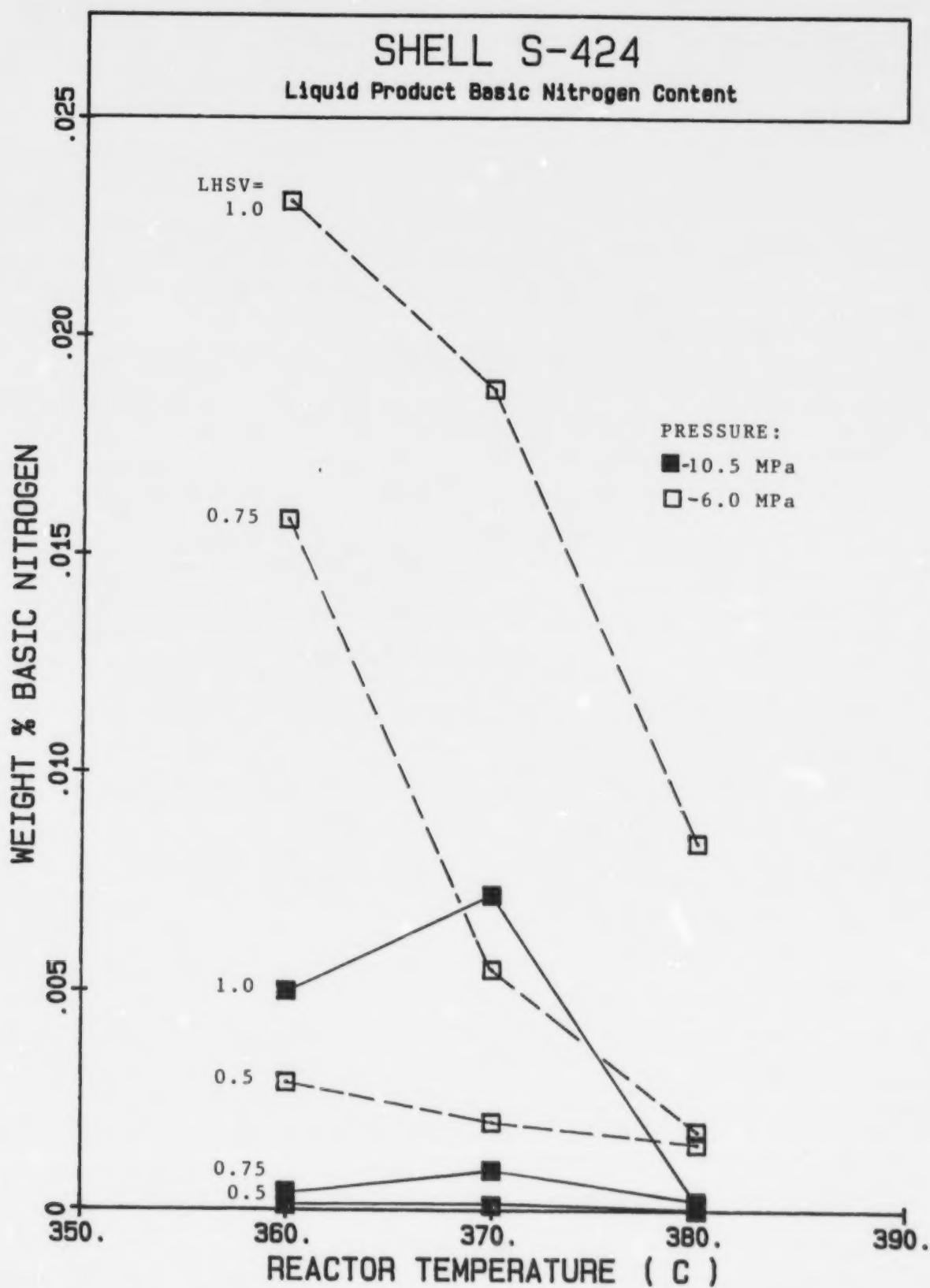


Figure B-13 - Liquid Product Basic Nitrogen Content - S-424

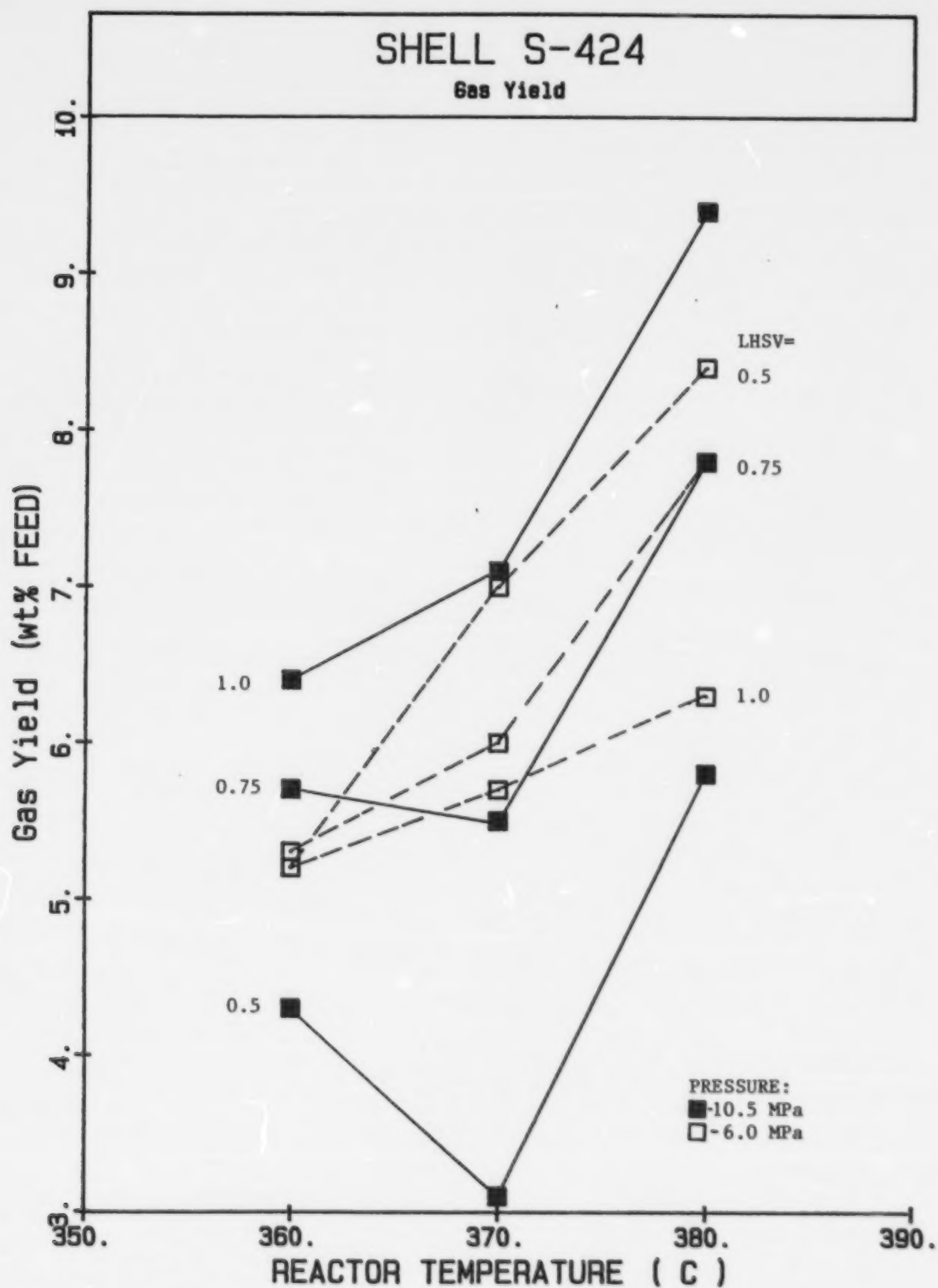


Figure B-14 - Gas Yield - S-424

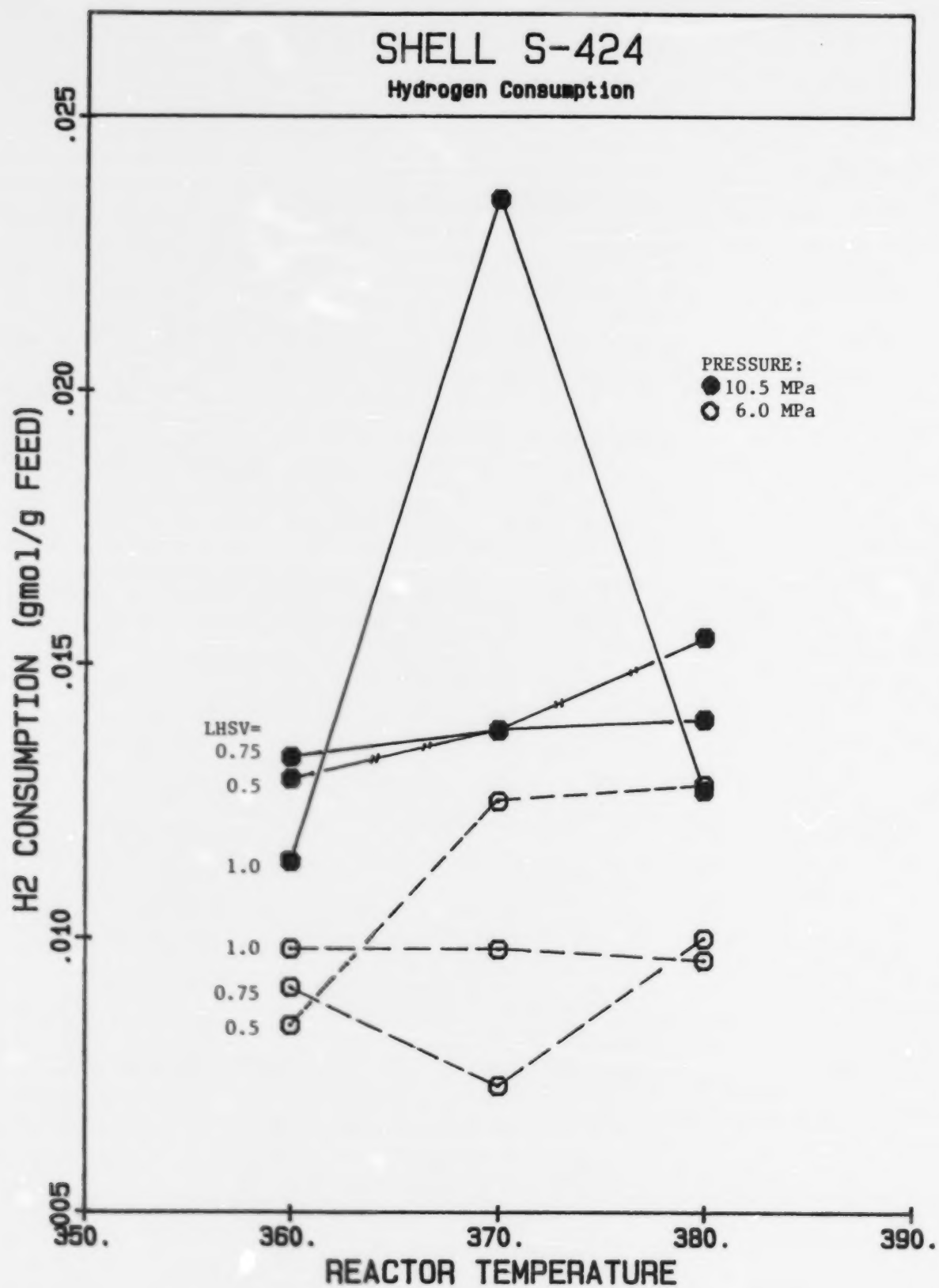


Figure B-15 - Hydrogen Consumption - S-424

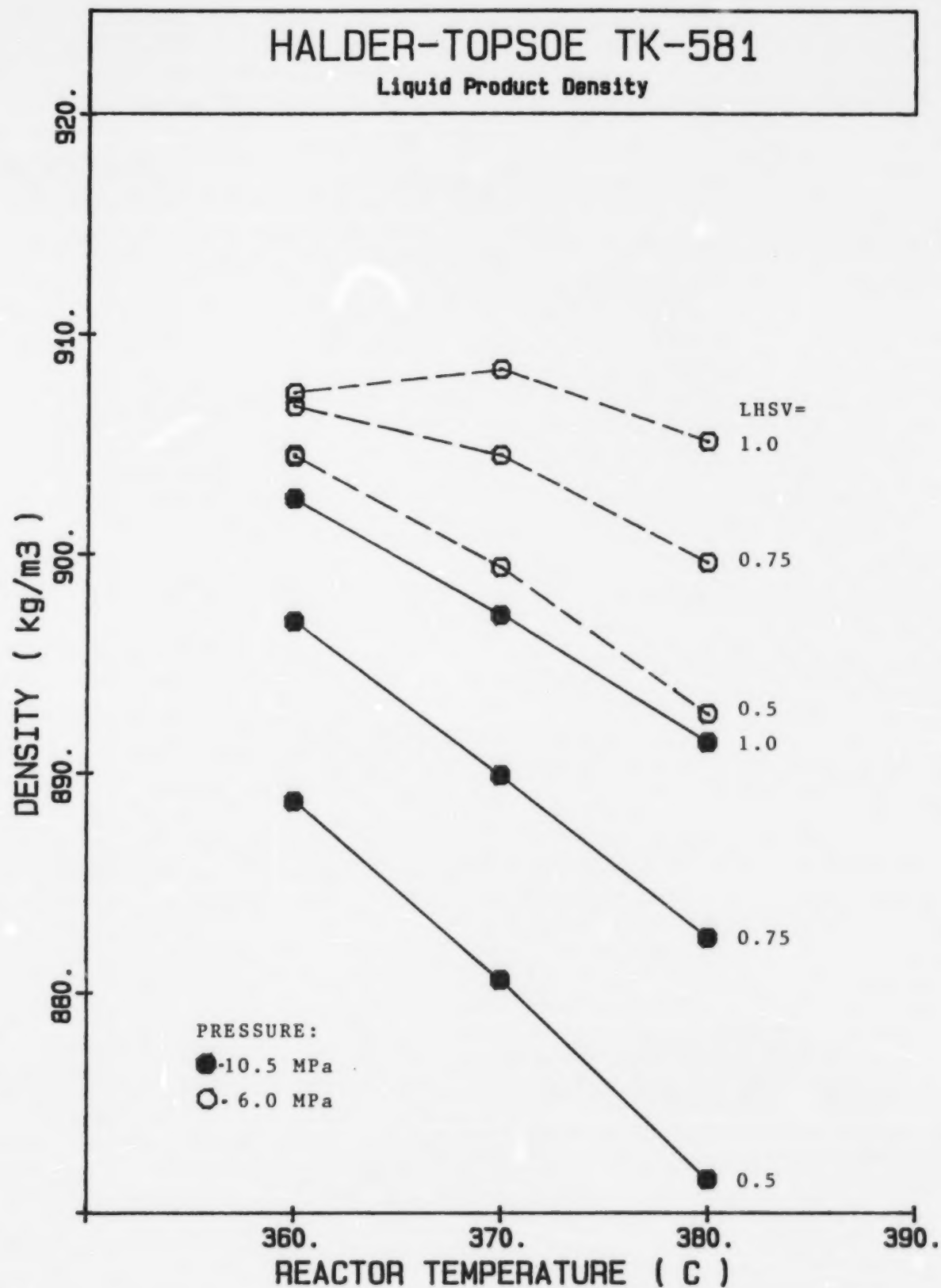


Figure B-16 - Liquid Product Density - Halder-Topsoe TK-581

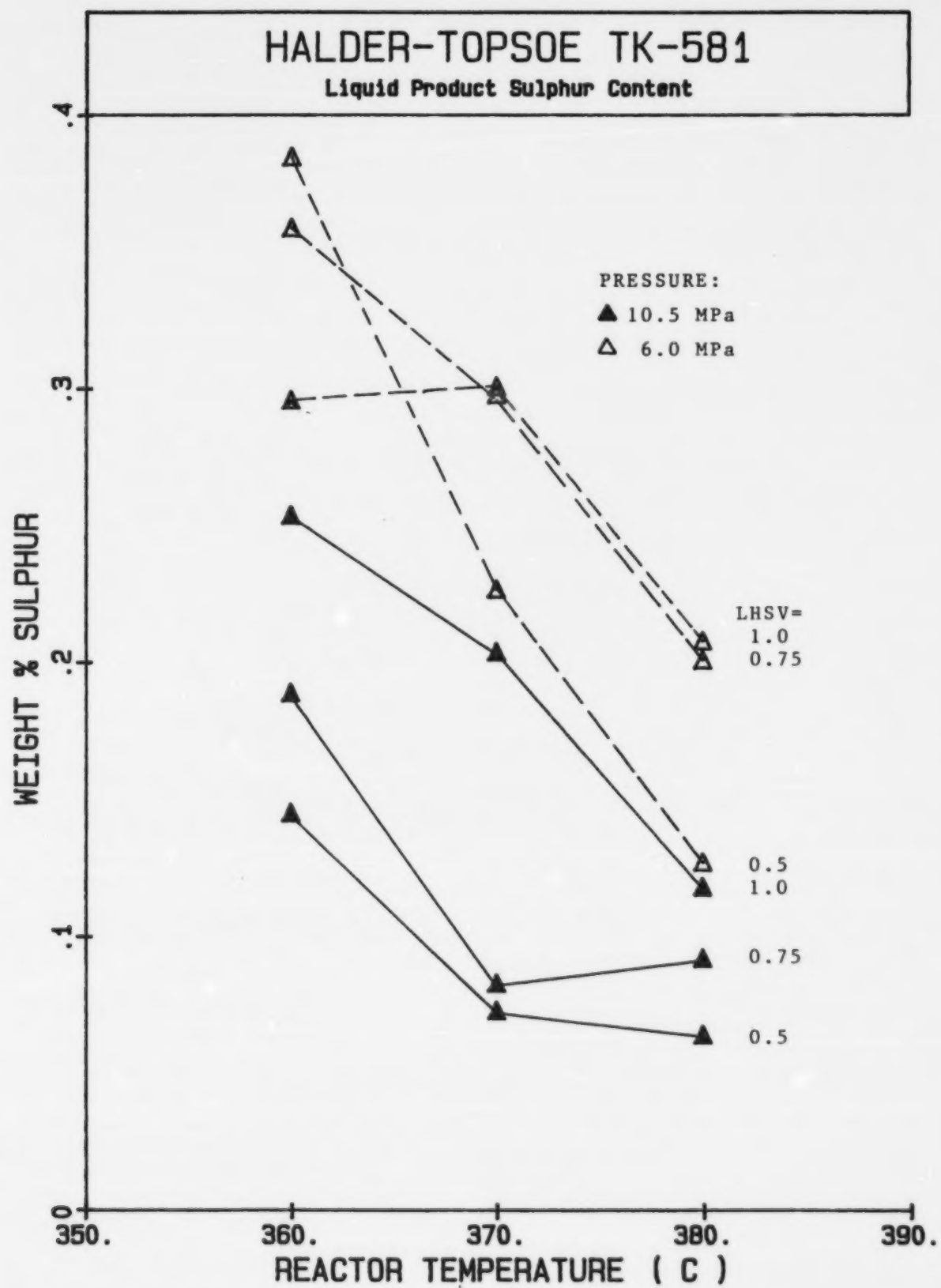


Figure B-17 - Liquid Product Sulphur Content - TK-581

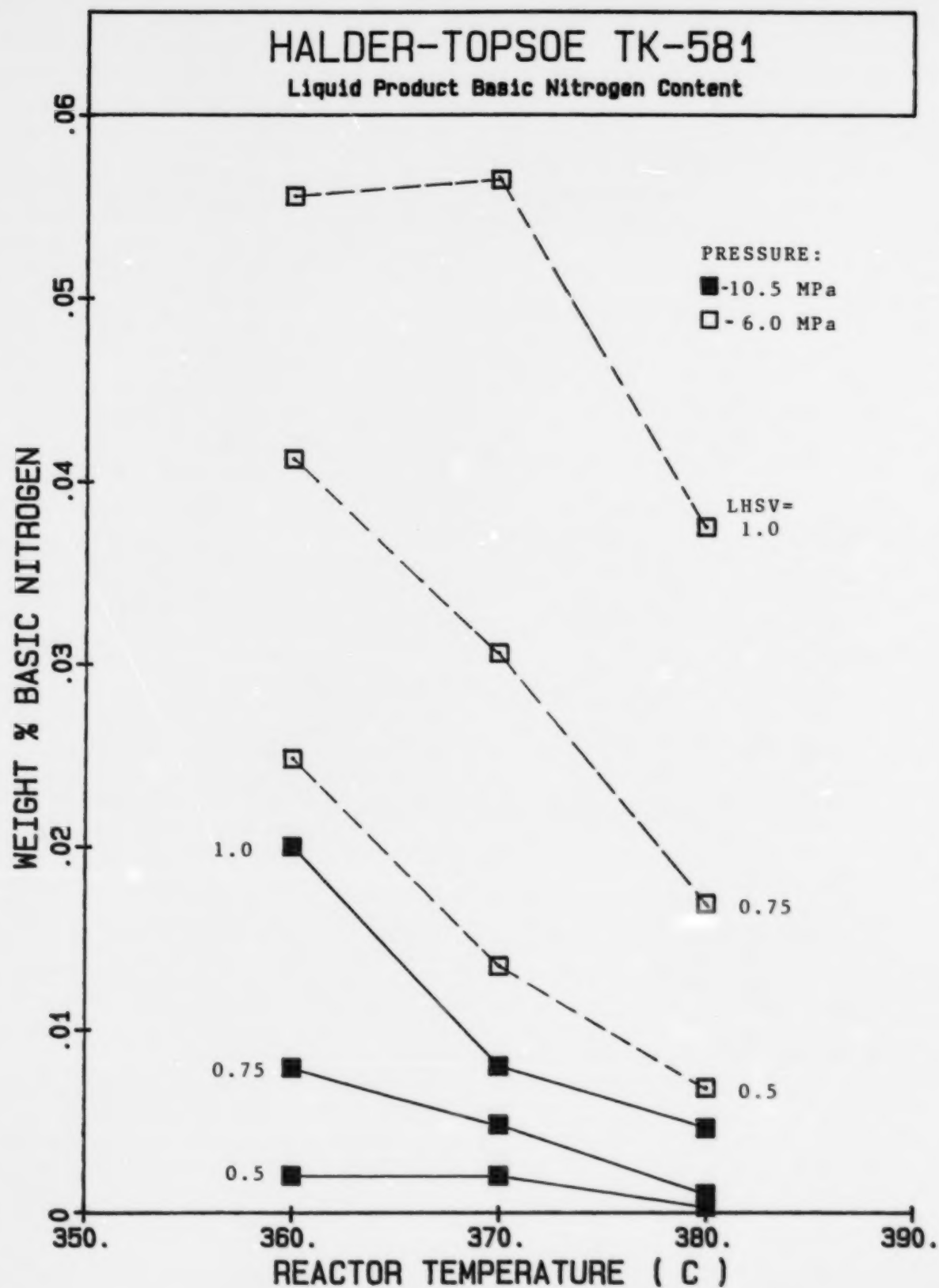


Figure B-18 - Liquid Product Basic Nitrogen Content - TK-581

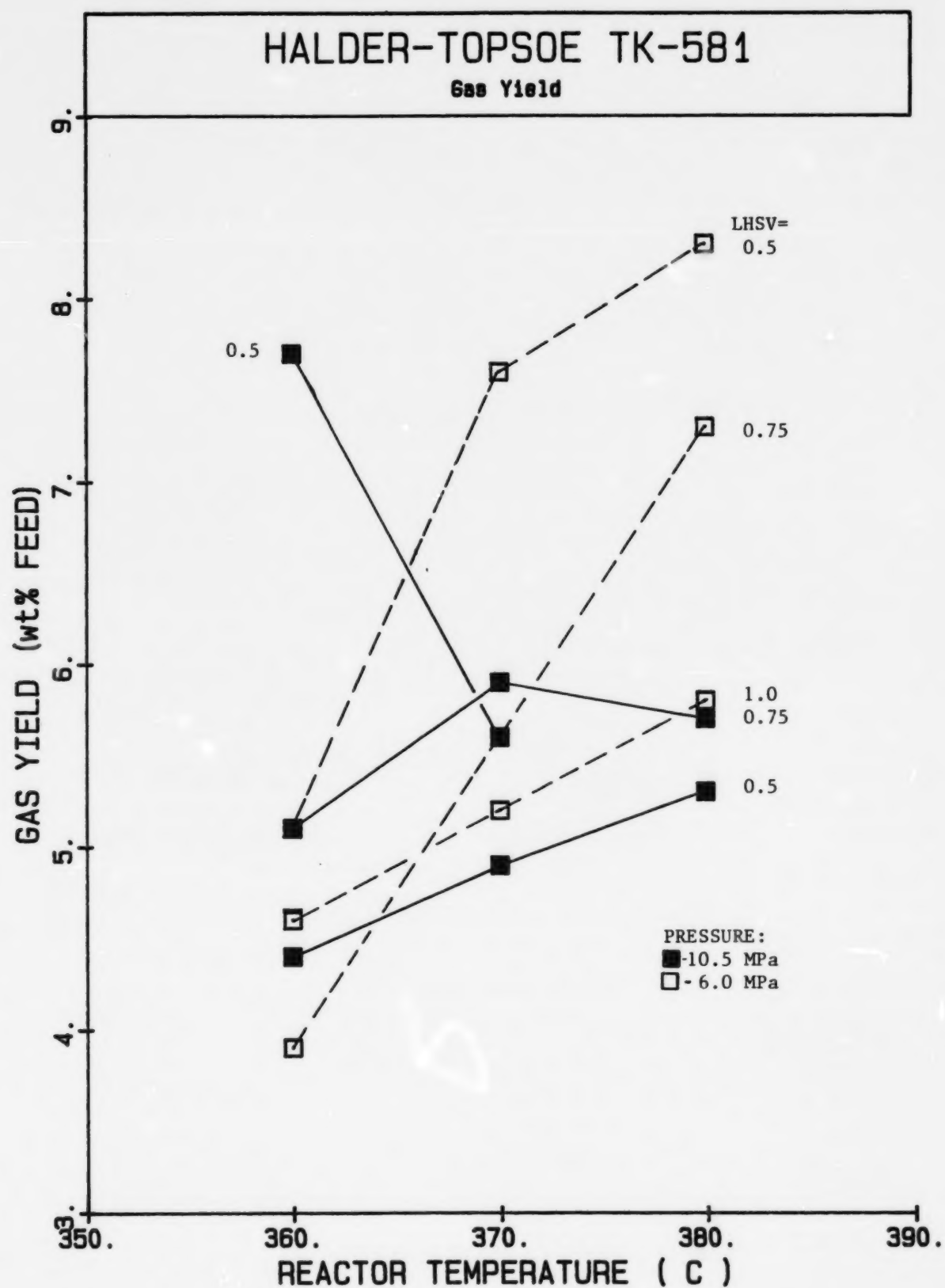


Figure B-19 - Gas Yield - TK-581

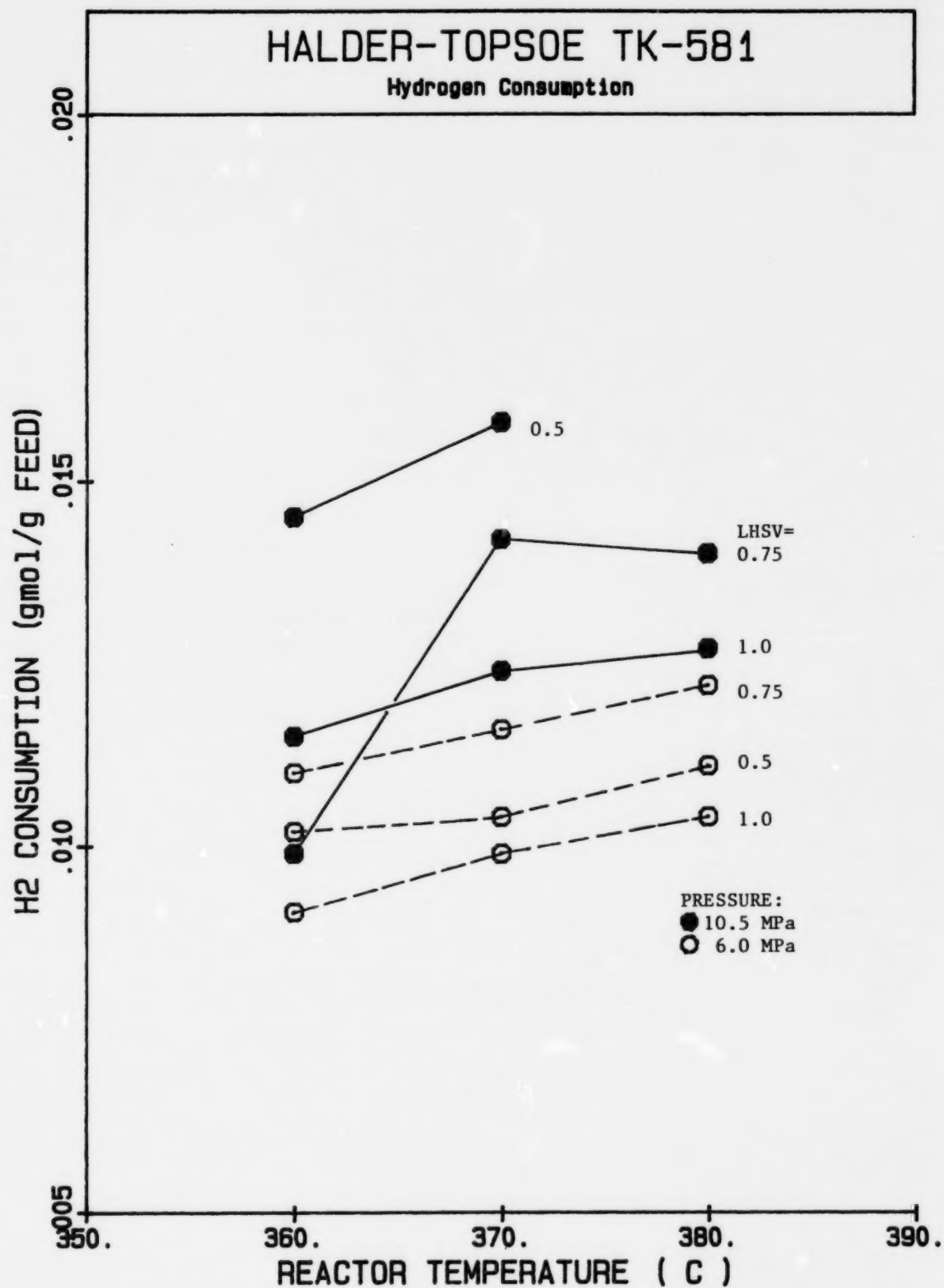


Figure B-20 - Hydrogen Consumption - TK-581

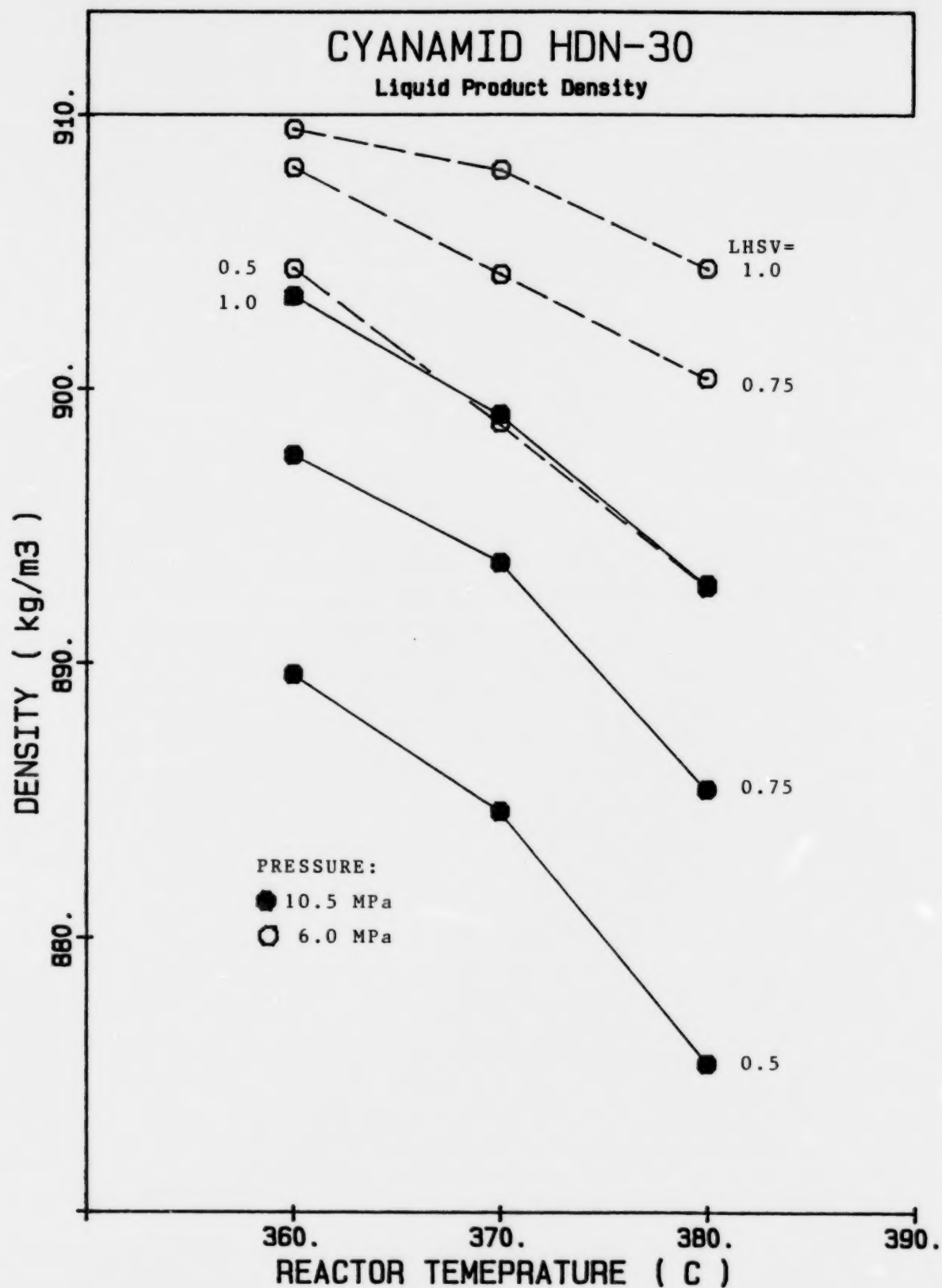


Figure B-21 - Liquid Product Density - Cyanamid HDN-30

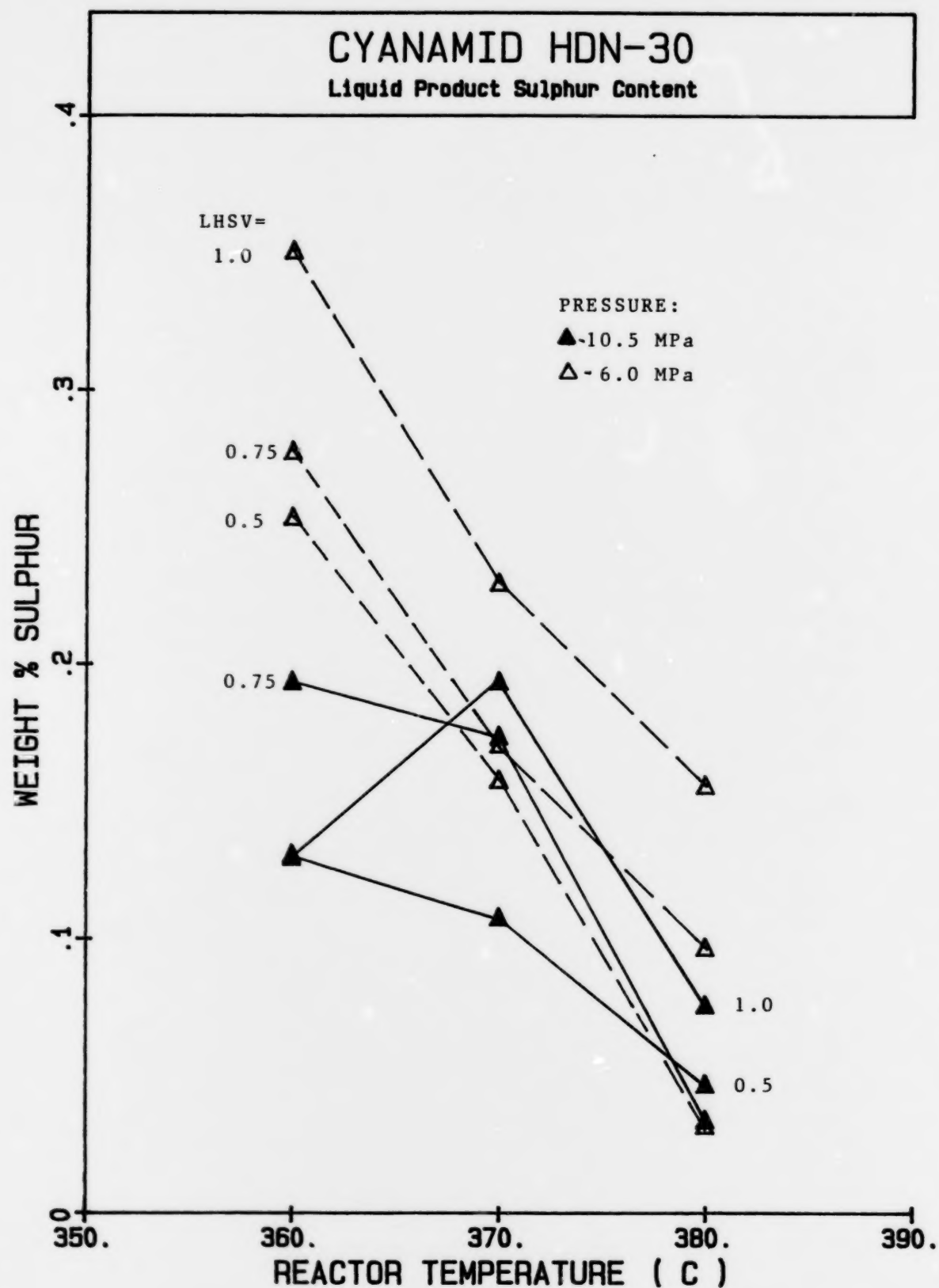


Figure B-22 - Liquid Product Sulphur Content - HDN-30

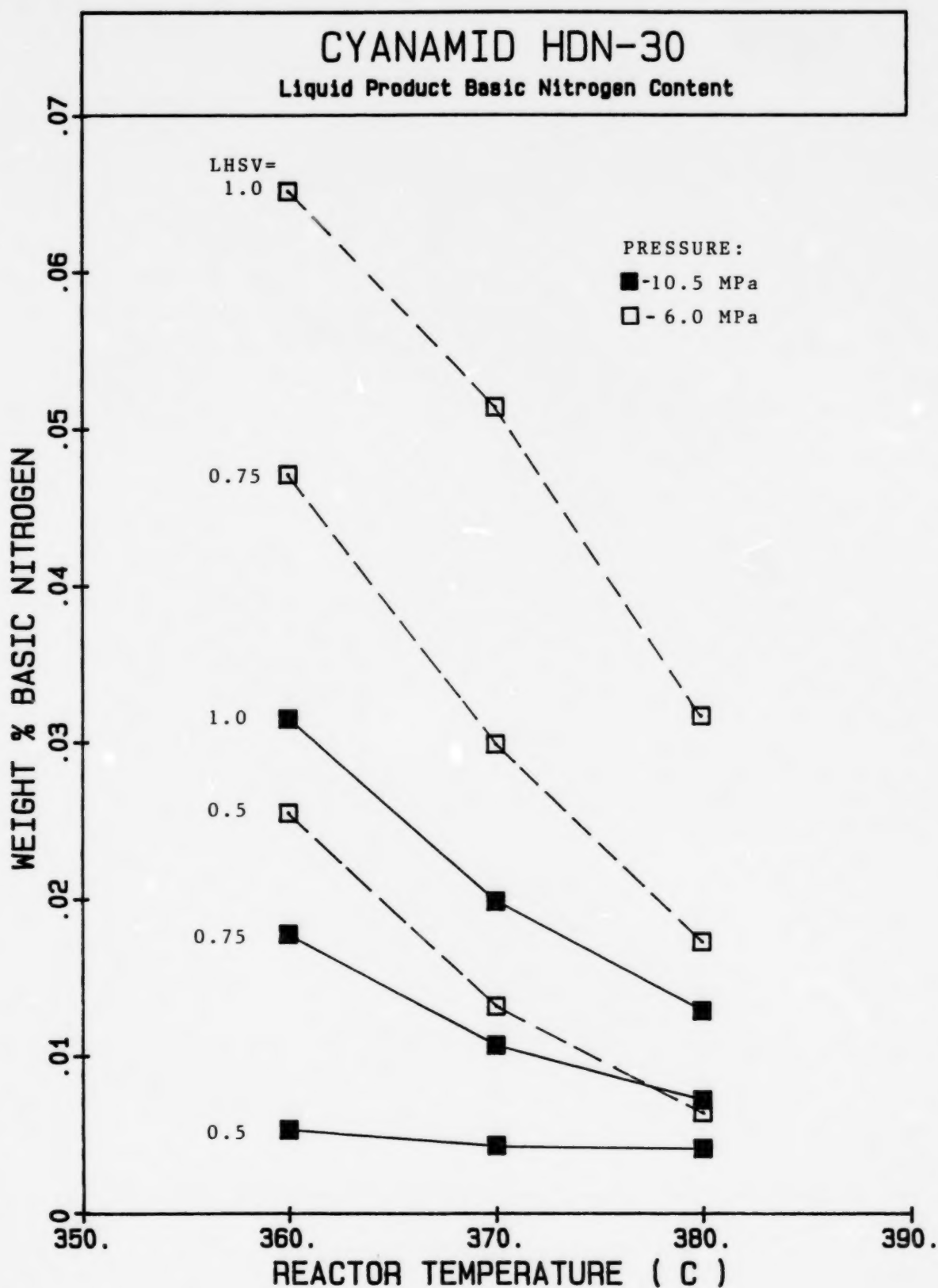


Figure B-23 - Liquid Product Basic Nitrogen - HDN-30

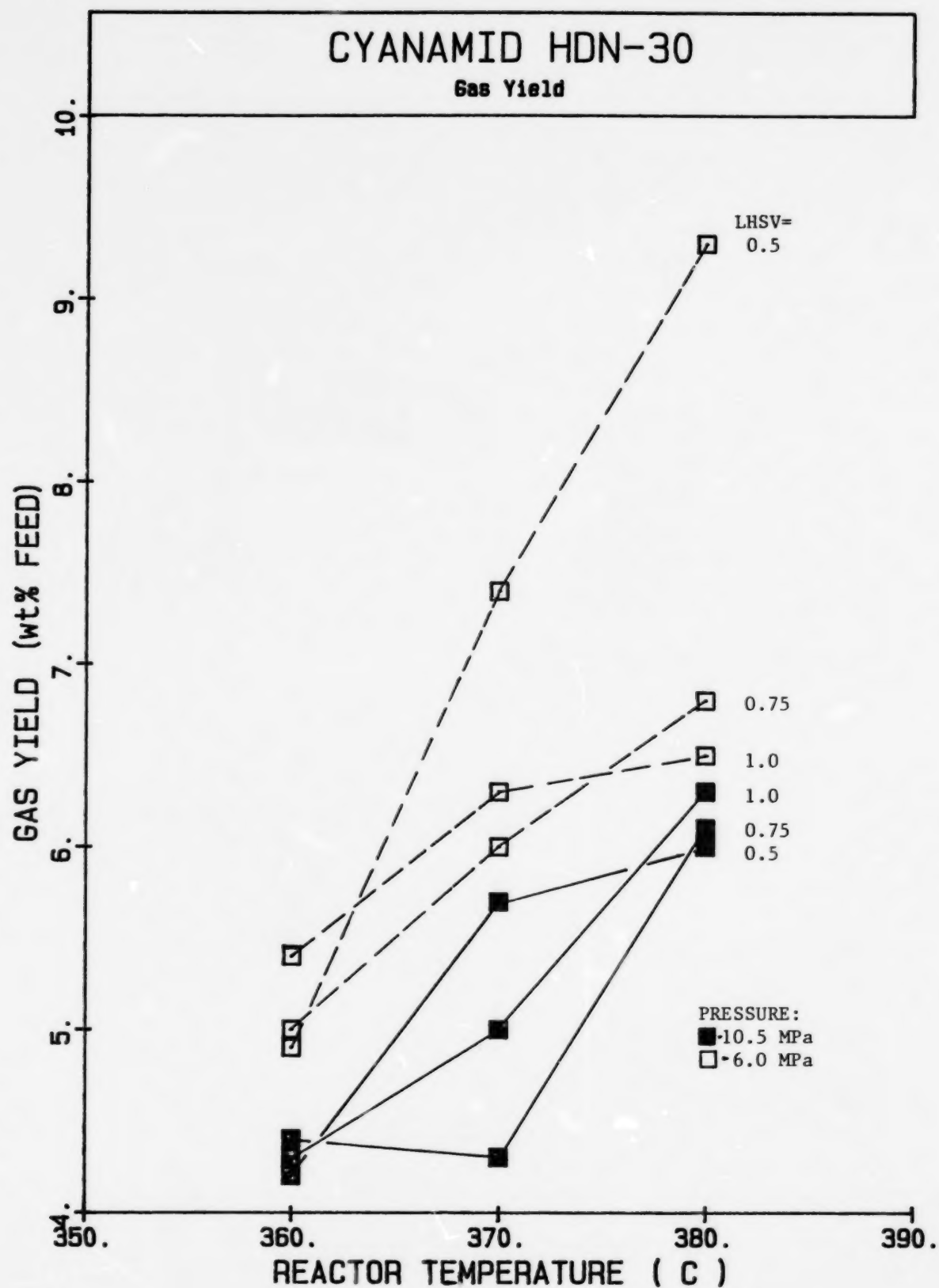


Figure B-24 - Gas Yield - HDN-30

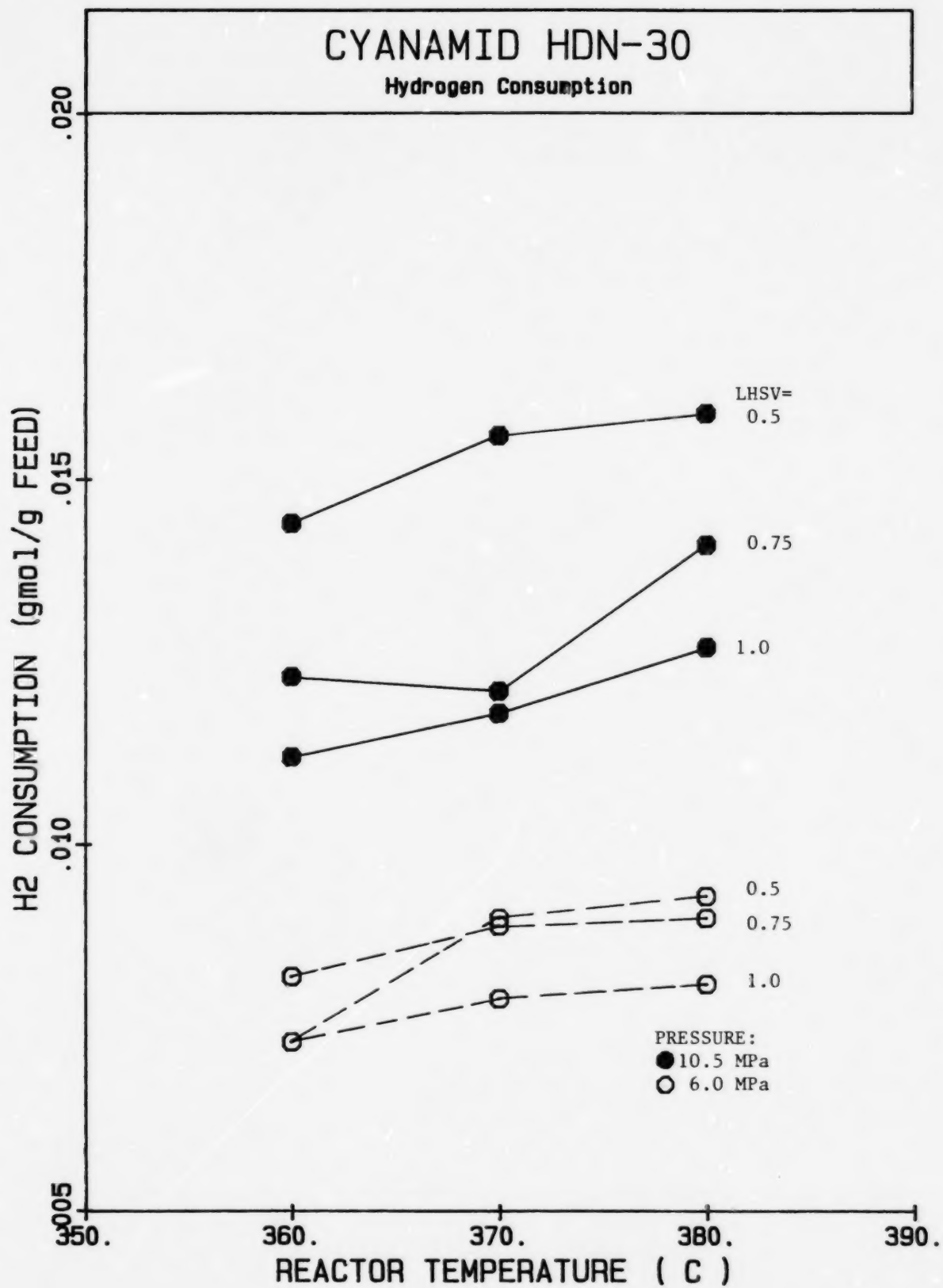


Figure B-25 - Hydrogen Consumption HDN-30

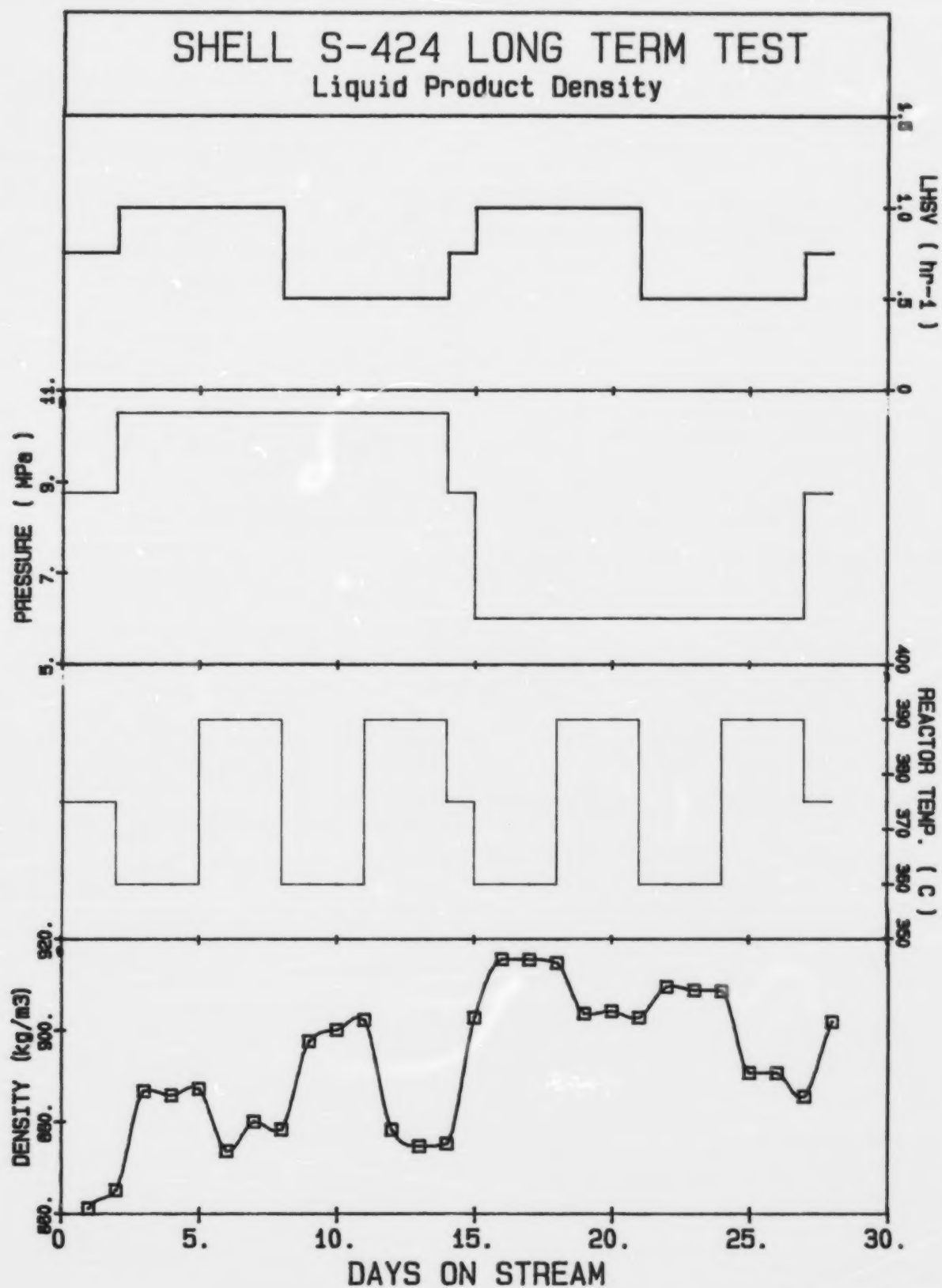


Figure B-26 - Liquid Product Density - Long-Term Test

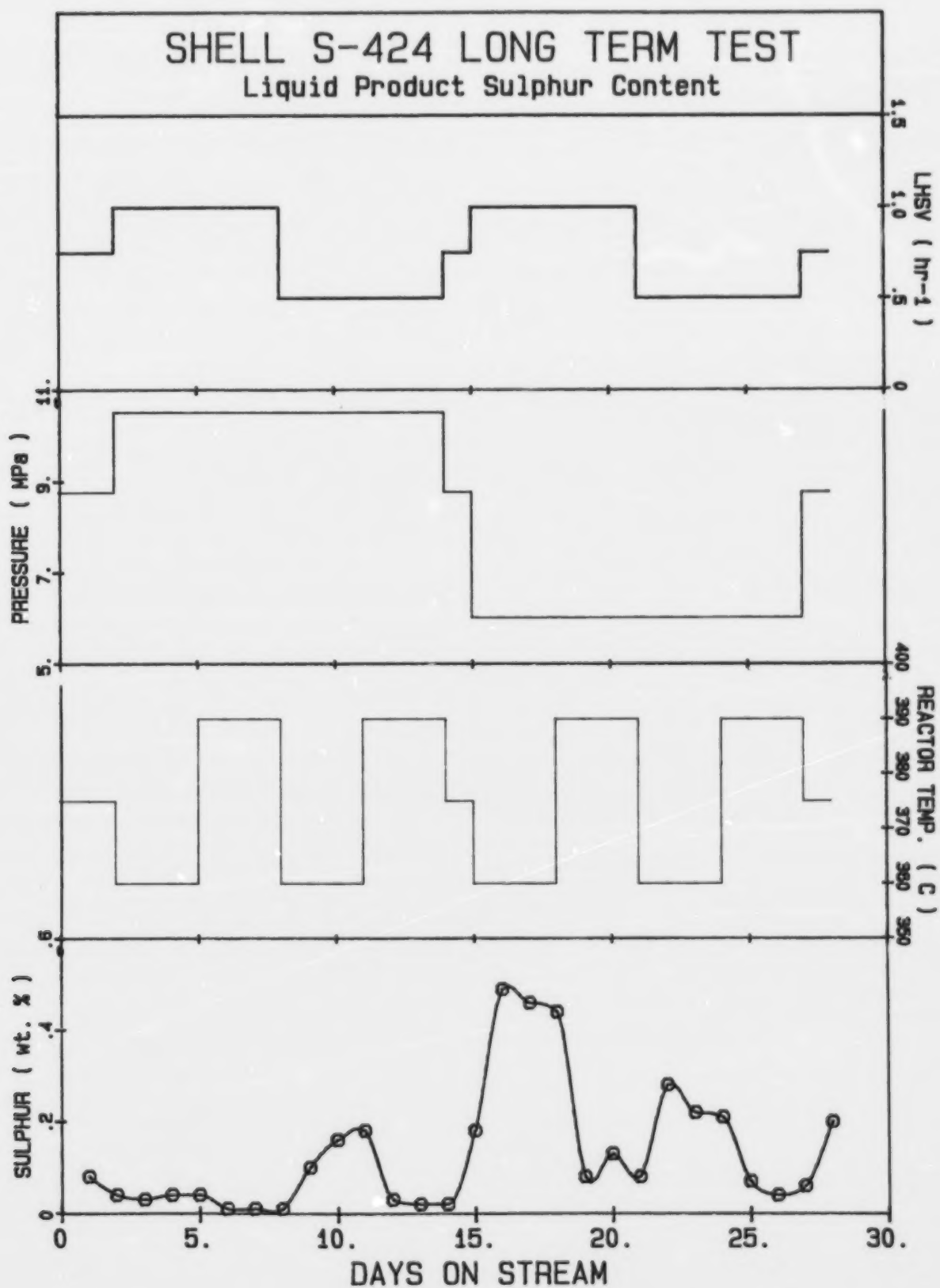


Figure B-27 - Liquid Product Sulphur Content - Long-Term Test

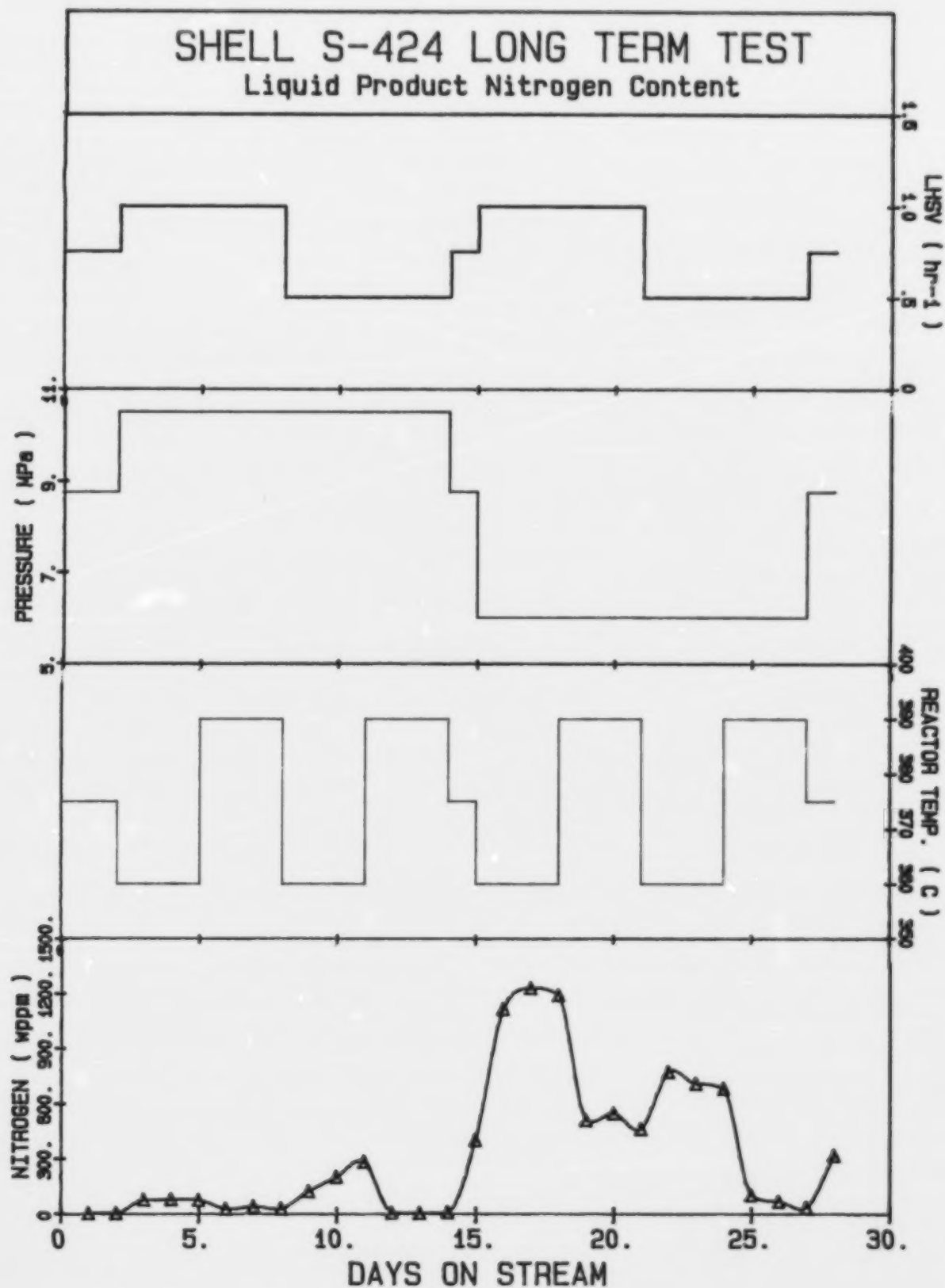


Figure B-28 - Liquid Product Nitrogen Content - Long-Term Test

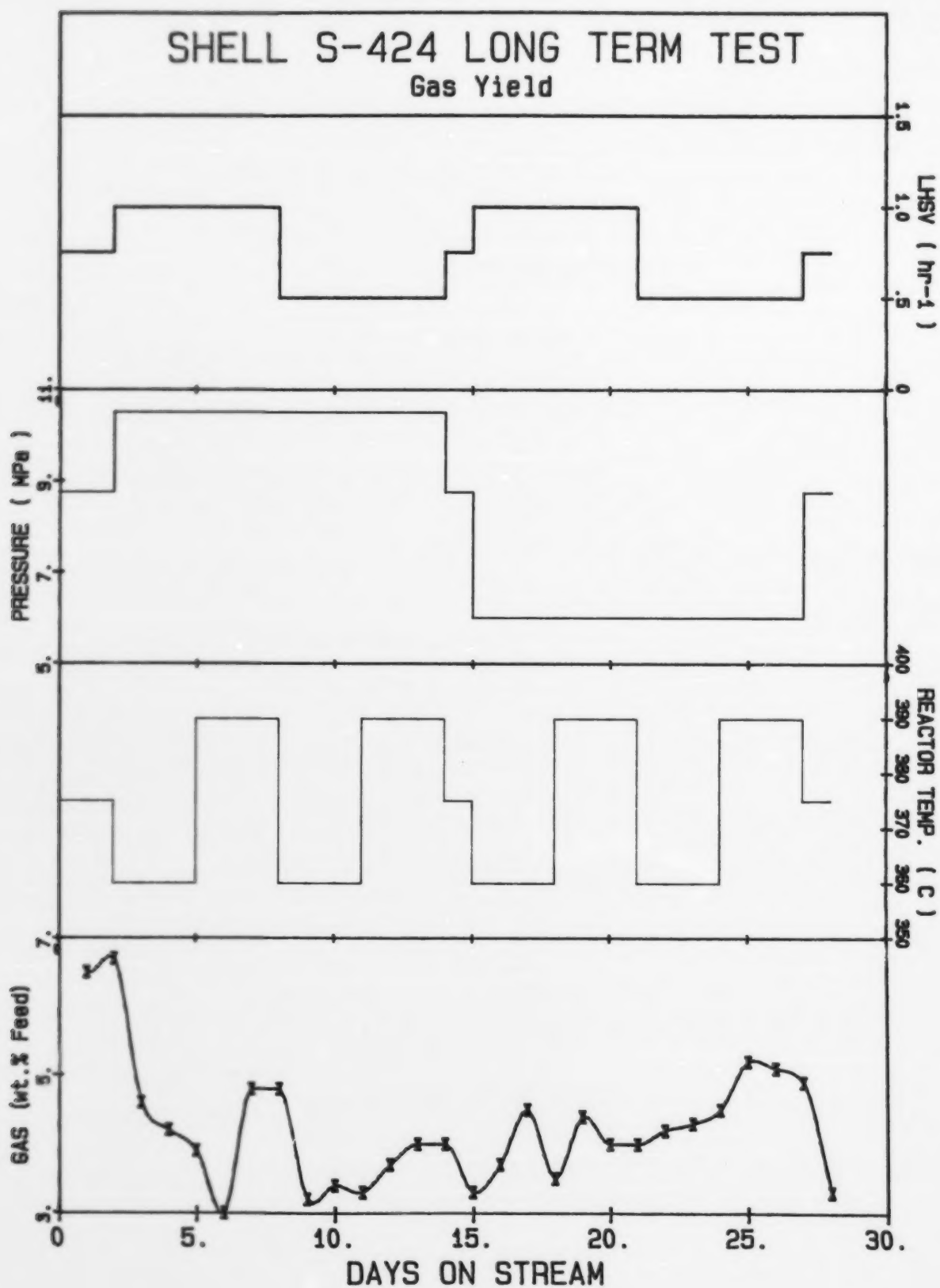


Figure B-29 - Gas Yield - Long-Term Test

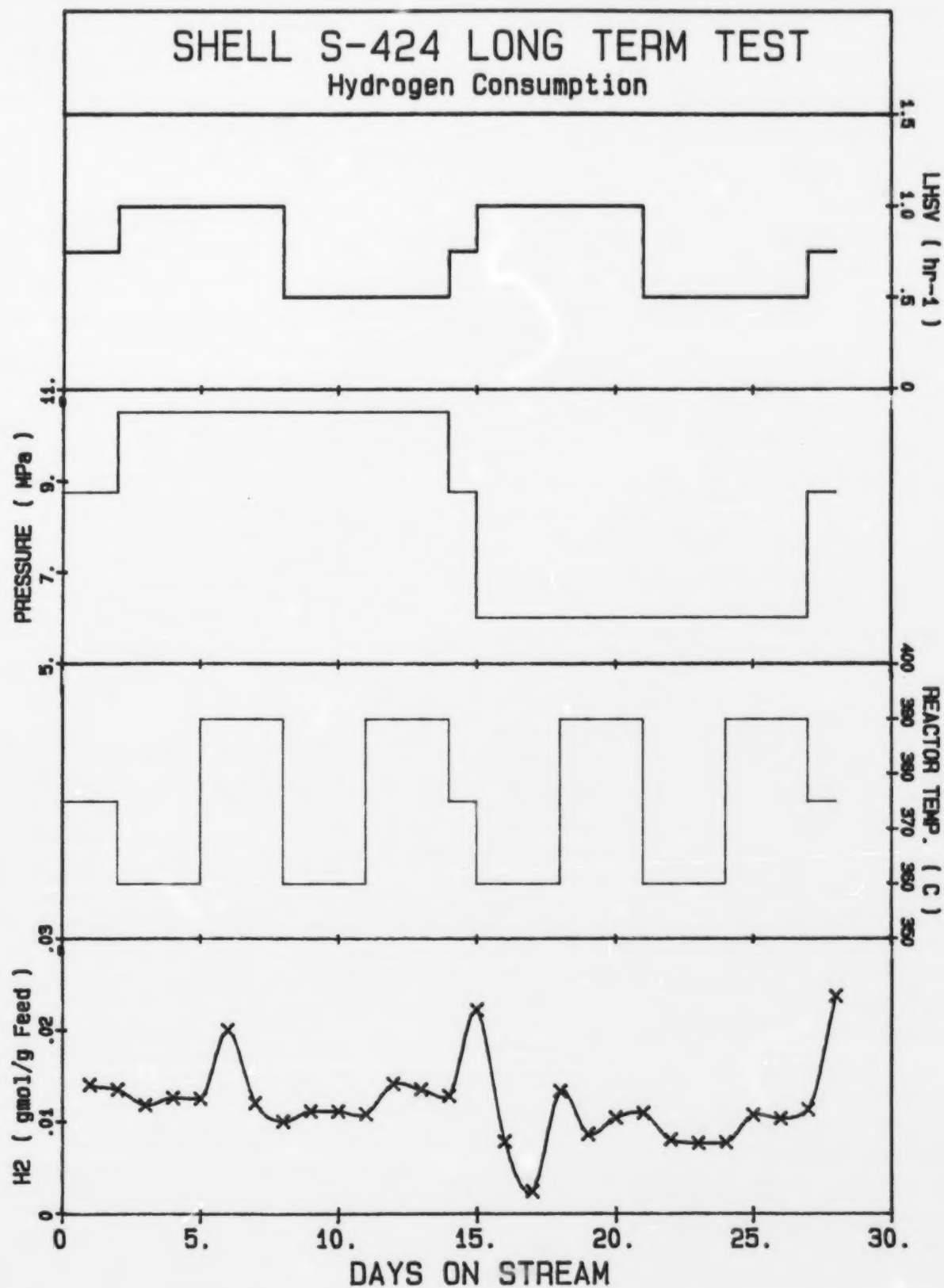


Figure B-30 - Hydrogen Consumption - Long-Term Test

APPENDIX C

PRESULPHIDING PROCEDURE - LONG-TERM TEST

Long-Term Test Presulphiding Procedure

1. Ambient pressure nitrogen purge for 30 minutes at 100°C.
2. High pressure (290 psig or 2.0 MPa) hydrogen purge for 30 minutes.
3. Presulphiding feed, light gas oil spiked with 2.5 wt % carbon disulphide (CS_2), feed at LHSV = 5.33 hr^{-1} and pressure at MPa (435 psig), no hydrogen flow. Maintain this rate for 3 hours.
4. Reduce feedrate to LHSV = 1.33, set hydrogen feed rate to 79 mL H_2/mL feed and proceed according to the following temperature rate:

	<u>Temp (°C)</u>	<u>Temp (°C)</u>	<u>Hold for Hours</u>
i)	100	250	5
ii)	250	300	5
iii)	300	320	6

5. Change to test feed and go to test conditions.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC)



III. CANADIAN HYDROTREATING TEST CENTRE (CHTC)

The Canadian Hydrotreating Test Centre (CHTC) was conceptualized in 1984 when there existed a growing demand for testing

- hydrotreatability of various feedstocks that are potentially the intermediate streams in the primary or secondary upgrading processes and,
- performance and hence optimal utilization of commercially available hydrotreating catalysts.

Selection of the physical site for the CHTC shall consider

- technical capability of the organization with which the finished CHTC will have a custodian association
- availability of supporting facilities, and
- location with respect to population of prospective users of CHTC.

Petroleum Division of Saskatchewan Research Council (Regina, Saskatchewan) is situated right in the areas of heavy oil industry, has available a High Pressure Research Laboratory for CHTC installation and is a reputed hydroprocessing research organization. It is therefore the logical choice for the CHTC site.

1. OBJECTIVES

The primary objectives for the Canadian Hydrotreating Test Centre are:

- a. Hydrotreatability Test for a variety of feedstocks ranging from naphtha, distillates, gas oils to heavy residues.
- b. Performance Test for commercial and proprietary hydrotreating catalysts.
- c. Process Study for the above a. and b.

It is expected that Desulphurization, Denitrogenation (especially reduction of the basic nitrogen) and Demetallization will be the key criteria for evaluating hydrotreatability. In addition, catalyst performance will be assessed against its activity decay. Given sufficient experimentation and analyses, kinetics of catalytic hydrotreating reaction may also be studied.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

1. OBJECTIVES (cont'd)

The CHTC with a size approaching to a demonstration plant will enable the industrial clients to extract valuable information such as:

- o mass flux along hydrotreating reactors, or space velocity
- o operating pressure, total pressure and partial pressure of hydrogen
- o various temperatures and temperature profile in the system
- o system thermal hydraulics
- o hydrogen consumption

for a process study that leads to commercial plant design and optimization.

The objectives as stated form the basis on which the process and facility systems are designed.

2. DESIGN BASIS

The basis of CHTC process systems and facilities design is formulated next. More detailed design will be presented in Section 3 and the subsequent write-ups.

2.1 CHTC Plant Throughput

As CHTC is to serve not only the R & D functions as stated in Objective a and b, but also the industrial development oriented Objective c (Section I.1 Objective) for the corporate users, the throughput should be within the range of scale-up to a demonstration plant. Using a scale-up factor of 50 and a throughput for a hydrotreating demonstration plant at 50 BBL/day, CHTC should be designed for a throughput of approximately 1 BBL/D. That is, a feedrate of approximately

$$F = 6 \text{ kg/hr.}$$

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

2. DESIGN BASIS (cont'd)

2.2 Hydrotreating Reactor Sizing

A competing factor must also be considered. This is the MASS FLUX along the hydrotreating reactor. For a Trickle Flow Reactor or a downward Plug Flow Reactor, a general guideline is

$$\dot{M} = \text{mass flux} = 4 \text{ kg/m}^2 \text{ sec.}$$

which, if adopted, would translate to a reactor cross sectional area of, roughly,

$$A = F/\dot{M} = 4.2 \times 10^{-4} \text{ m}^2 = 4.2 \text{ cm}^2$$

Experience shows that the Liquid Hour Space Velocity (LHSV) falls in the range of

$$\begin{aligned} \text{LHSV (hr}^{-1}\text{)} &= 0.5 - 1.0, \text{ Heavy Feedstock} \\ &= 1.0 - 2.0, \text{ Light Feedstock} \end{aligned}$$

Conveniently, an LHSV=1 is adopted as the sizing basis. This yields an approximate volume V:

$$V = F/\text{LHSV} = 6 \text{ kg Equivalent volume} = 6 \text{ litres}$$

Such a reactor would have a diameter of 1" and a height of 40', an unrealistic configuration. A design compromise is taken, resulting in a sectioned reactor of 3 litres total volume with a 1" nominal diameter and 20' overall height and a mass flux of, at LHSV=1,

$$\dot{M} = 1.73 \text{ kg/m}^2 \text{ sec.}, \text{ LHSV} = 1.0, F = 2.884 \text{ kg/hr}$$

For light feedstock, the corresponding values are

$$\begin{aligned} \dot{M} &= 3.5 \text{ kg/m}^2 \text{ sec.}, \text{ LHSV} = 2.0, F = 5.77 \text{ kg/hr} \\ & \quad (0.95 \text{ BBL/D}) \end{aligned}$$

Detailed design of the reactor assembly is presented later.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

2. DESIGN BASIS (cont'd)

2.3 Hydrogen Supply and Recycle

Hydrotreating test of Suncor Gas Oil indicated that hydrogen consumption ranged from 25 to 45% of the feed

$$\begin{aligned} G &= 0.0418 \text{ g-mole/g-liquid feed} \\ &= 0.1205 \text{ kg-mole/hr (Gas Oil F = 2.884 kg/hr)} \\ &= 2,700 \text{ sl/hr} \end{aligned}$$

For a once-through system, the unreacted, recoverable hydrogen could be very significant - roughly 1,800 sl/hr or some 60% of the feed gas. A Hydrogen Recycle Compressor is therefore required.

The size of Hydrogen Recycle Compressor, however, should cover the entire operating range of light to very heavy feedstock. For heavy hydrotreating feed, partial pressure of hydrogen and hydrogen demand are expected to be higher than those of the Suncor Gas Oil. On this basis, the Compressor is sized to 4,100 sl/hr at a polytropic head of 25 MPa max.

2.4 Run Time and System Residence Time

Both are factors affecting sizing of vessels and operation.

The System Residence Time is defined as:

$$t_s = \text{Sum of } t_{s,i}$$

$$t_{s,i} = \text{Unit Residence Time} = \frac{\text{Unit Volume}}{\text{Flowrate in the Unit}}$$

The Residence Time should be evaluated along the "Longest Route", which, in the present case, includes P-1 (Feed Pump) E-1, R-2, R-1, V-1 (liquid blowdown portion only), V-2 (liquid blowdown portion only), F-2 and the line runs along the flow path (cf. Appendix D). For an eight-hour per operating day, maximum t_s is set at

$$\begin{aligned} t_s &= 4 \text{ hrs. for LHSV} = 1.0/\text{hr} \\ &= 8.0 \text{ hrs, approx. for LHSV} = 2.0/\text{hr.} \end{aligned}$$

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

2. DESIGN BASIS (cont'd)

The run time per batch feeds, on the other hand, is set to enable a continuous experimentation for the key parameters involved, that is:

Pressure:	3 values (min. 2 values)
Temperature:	3 values (min. 2 values)
LHSV:	2 values (max. 3 values)

Total:	6 runs (min.) to 27 runs (max.) per catalyst loading
--------	--

Based on a system constant of $t_s = 4$ hrs. plus a steady run time of 6 hrs., a total of 10 hrs. would be required per set of parameters. Hence for a catalyst loading, total experimentation run would take from $6 \times 10 = 60$ hrs. to $27 \times 10 = 270$ hrs. approximately. Feed Tank and hydrotreated Product Storage Tank should therefore be sized to, for LHSV = 1, $F = 3$ l/hr.

$$\text{Volume} = F \times 270 \text{ hrs} = 3 \times 270 = 810 \text{ l}$$

3. SYSTEM DESIGN DESCRIPTION

(cf. PFD Drawing No. 080-B3000, Appendix C and P & ID Drawing No. 080-D3001, Appendix D)

The Process Flow Diagram (PFD) shows, in a simple manner, the essential system configuration. The Material Balance Table at the bottom half of the PFD was established for LHSV = 1.0, using the hydrotreating test results of this project as a calculation base. The Design Basis depicted in Section 2 was adopted in developing the PFD and the Piping and Instrumentation Diagram (P & ID).

3.1 Liquid Feed System

This subsystem consists of receiving, storage, transfer and feed control of the liquid hydrotreater feedstock.

The virgin feedstock which comes in as drums or, most probably standard oil barrels (42 US gallons/barrel), will be pumped via a flexible hose which is coupled, connected at Feed Tank (T-1) nozzle. The receiving barrel pad and barrel transfer pump are described in the next Section 4.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

3. SYSTEM DESIGN DESCRIPTION (cont'd)

Feed Tank (T-1), a horizontal, atmospheric vessel, has a capacity of 830 l minimum which meets the design based value of 810 l (Section 2.4). The tank is equipped with an internal electrical heater (Feed Immersion Heater H-3) which acts as feed preheater and, in case of heavy feedstock of high pour point, serves to reduce the viscosity (increasing pumpability).

Feed Pump (P-1) can be either a plunger or a diaphragm type. Relief or minimum flow recycle to Feed Tank is required, either by internal design or by an external arrangement such as shown in the P & ID. Owing to wide variation of feedrate (LHSV from 0.5 to 2.0), a variable speed pump is recommended. Automatic feed flow control can be achieved either by (pneumatic or electrical) stroke adjustment or by an external flow control loop as shown. To minimize start-up transient time, a pump start-up loop is designed.

The liquid feed line joins hydrogen gas feed line. Piping upstream of the main Hydrotreating Reactor (R-1) requires tracing (electrical or steam) and insulation.

3.2 Hydrogen Make-up and Recycle

Feed hydrogen consists of two streams: Recycle from H.P. Separator (V-1) and Make-up from hydrogen cylinder. Recycled hydrogen is sent back through a pressure control valve (PCV) to the hydrogen feed header. This stream contains between 0.5 to 1.0% of light hydrocarbons (methane and ethane, mostly). Depending on the recycle rate, hydrogen make-up rate is controlled by a flow control loop, to maintain a fixed H_2 /liquid feed ratio. A Hydrogen Compressor (C-1) is installed to deliver the gas to the required system pressure.

Because of recycling, the combined hydrogen feed stream will see gradual accumulation of hydrocarbon impurity. This would result in a significant reduction in hydrogen partial pressure in the reactor, over a prolonged period of "solid mode operation". To prevent this, occasional manual bleed, via a bleed valve on H.P. Separator, should be made. Decision for bleeding is to be taken from sample analysis result (Note: a recycle hydrogen sample connection on the recycle line).

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

3. SYSTEM DESIGN DESCRIPTION (cont'd)

3.3 Reactor System

The combined hydrogen and liquid feed, being preheated by Interchanger (E-1) can be fed either directly to Hydrotreating Reactor (R-1) or, in sequence, to Pretreating (Guard) Reactor (R-2) then to Hydrotreating Reactor.

Utilization of Pretreating Reactor depends on requirements to demetallize virgin feedstock, either for downstream processing need (such as for Fluid Catalytic Cracker, FCC operation) or to prevent hydrotreating catalyst poisoning. Note that pretreating catalyst is different from that used in the main hydrotreating reactor.

Hydrotreating is generally exothermic. Reaction zone cooling/quenching (notably by make-up hydrogen) is usually desired. This can be incorporated in detailed design.

The two downwards, trickle bed reactors are insulated and equipped with Induction Heaters (H-1 and H-2). They are designed primarily for start-up pre-heating of reactors. But they can also be used for temperature programming when hydrotreating temperature parameter is changed for the next run.

3.4 Reactor Product Cooling and Interchanger

The hydrotreated reactor product, a mixture of

- unreacted hydrogen gas
- methane, ethane, propane and butane in a pseudo-continuous dense phase
- hydrocarbon liquid
- hydrogenation gases, primarily H_2S and N_2 , with H_2S appearing as a dense-phase fluid

should be cooled for heat recovery and, more importantly in the case of gas recycling for condensing H_2S and light hydrocarbons as much as possible.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

3. SYSTEM DESIGN DESCRIPTION (cont'd)

Reaction heat is partially recovered by transferring the energy to hydrogen and liquid charge mixture in the Interchanger (E-1). A temperature drop from 390°C to 220°C is expected, which would give the feed a corresponding temperature rise from 65°C to 190°C.

At 220°C and system pressure of, say, 10 MPa, further cooling of reactor product is needed. This is accomplished by a water-cooled heat exchanger, Product Cooler (E-2). The temperature is taken down to 80°C or less, below the dewpoint of H₂S and C₃ (propane).

Owing to small thermal duty involved, both E-1 and E-2 can be constructed as double-pipe exchangers.

3.5 H.P. and L.P. Products Separation

The cooled high pressure (H.P.) product flows to H.P. Separator (V-1) vessel. The now clearly distinctive gas and liquid products are separated out, with gases rising in the upper space and liquid settling in the bottom. Through a back pressure control valve (self-actuated), gases are let down to 210 kPa or less as recycle stream to the suction of Hydrogen Compressor (C-1). Accumulated liquid will be let down via a Level Control Valve. Liquid is flashed to L.P. (low pressure) Separator (V-2).

The flashing two-phase fluid enters L.P. Separator, with H₂S, NH₃ (small quantity), C₂, C₃, C₄ and C₅ in the gas phase which is subsequently flowing to Gas Product Treatment System. The hydrotreated liquid product is let down via a L.P. Level Control Valve to the Liquid Product System.

An in-line Liquid Product Filter (F-2) is installed upstream of L.P. Level Control Valve. Entrained solids of a size 5 microns and larger will be retained. Its downstream Flow Element (FE) (or flow meter) is thus protected.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

3. SYSTEM DESIGN DESCRIPTION (cont'd)

3.6 Gas Product System

Hydrotreated gas product contains sulphides (H_2S and minute amount of carbonyl sulphides, depending on severity of treating) is scrubbed off in Caustic Scrubber (V-3). The scrubber, a vertical vessel, is filled with a caustic solution which reacts with sulphide ions S^{2-} and HS^- . De-sulphided gas stream should be dried to get rid of entrained solution and water mists. This is the function of Gas Dryers (S-1 and S-S, spare/standby). Dried gas is further subject to filtration in Gas Product Filter (F-1) which also prevents dryer bed solids from being carried over to the downstream atmospherical pressure (back-pressure control) letdown valve and dry- tested gas meter/gas flow element.

Treated gas discharging from meter is vented outdoor.

3.7 Hydrotreated Liquid Product Storage, Transfer and Recycle

Hydrotreated liquid product is provided with a Storage Tank (T-2) which is identical in size and design to the Feed Tank (T-1). Tank Immersion Heater (H-4) serves the same purpose as H-2 in T-1, since, in general, pour point improvement by hydrotreating is very marginal (although viscosity may be reduced to a greater extent).

The Product Transfer Pump, of a similar design to Feed Pump (P-1) should be sized up, according to processing requirement of downstream units such as FCC, stabilizer and so on.

An "off-spec" recycle line, sloped from P-2 suction to P-1 suction, is installed to enhance experimentability of the system.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

4. FACILITY DESIGN

The hydrotreating system described in Section 3 includes process units only. The additional facilities that are required to make up the entire, functionable Canadian Hydrotreating Test Centre are discussed herein.

References should be made to Plot Plan, Drawing No. 140-B2001, attached as Appendix H. The complete CHTC is shown to comprise:

- Process System as presented in Section 5.3
- Gas Cylinders Supply (Enclosure)
- Barrel/Drum Loading and Storage
- MCC (Motor Control Centre) Structure and Control Equipment
- Laydown Area/Facilities, Presulphiding and Analytic Facilities

In addition, it also requires

- Building Heating and Ventilation
- Electrical Supply
- Utility Supply

which are not shown in the drawing, and will be discussed in the next several subsections.

Note that the layout plan is limited by the size and dimensions of the existing High Pressure Bay in Petroleum Division, Saskatchewan Research Council.

4.1 Process Equipment, Piping and Layout

The Process Equipment List, complete with design data, is attached as Appendix F.

Except for the short section of make-up hydrogen line, all piping is to be insulated, mainly for personnel protection. Lines containing hydrotreating feed and liquid product shall be traced and insulated. Tracing is required for start-up and purging the left-over heavy

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

4. FACILITY DESIGN (cont'd)

distillates/resid charges after an extended shutdown. Although hydraulically smaller line size can be used, consideration of construction such as line support has necessitated the use of 1" NPS size for the entire system (except for local connection). Piping material containing feed or products (liquid and gas) shall be designed to "sour specifications" to minimize hydrogen sulphide corrosion.

Vents, drains, purge and sample take-offs are as indicated on P & ID (Drawing 080-D3001).

Layout for the process equipment is as shown on Plot Plan (Drawing 140-B2001).

4.2 Instrumentation, Electrical and Control

It should be aware that CHTC, including building peripherals, shall be designed to Class I, Division I code requirements. Electrical and control equipment such as:

- Motors (P-1, P-2 and C-1)
- Starters (P-1, P-2 and C-1)
- Breakers
- Lighting Fixture
- Control Stations
- Heaters (H-1, H-2, H-3 and H-4)
- Electrical receptacus/outlets and conduit

for the process system shall be explosion-proof. Equipment inside MCC enclosure, e.g. Transformer, however, does not require explosion-proof.

The CHTC process plant will be fully instrumented and automatically controlled, except for manual sampling. Data acquisition, transmission, recording, display and manipulation are included in the design. Transmission is shown by way of 4-20 mA signal, with some pneumatic signals being connected by P/I converters. A DCS system and an IBM-AT complete with printer and suitable software would be a good choice to implement.

The emerging Fibre Optics technology can also be adopted, in replacing DCS and electronic data transmission. Discussion on this is to be left for the future detailed design phase.

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

4. FACILITY DESIGN (cont'd)

4.3 Building and Supporting Facilities

The non-process related facilities which would be required to complete the entire CHTC are briefly discussed below.

MCC Structure/Room

A nominal size of 12' x 14' should be adequate. The enclosure should have double door construction and a POSITIVE PRESSURE supply to meet Class I, Division 1 stipulation. A concrete block base with glass windows above is felt the most convenient design. Pressurization can be supplied from building Heating and Ventilation System.

Building Heating and Ventilation (H & V)

An active, once-through ventilation shall be required. Design of H & V should consist of:

- a nominal 6 ACPH (air changes per hour)
- exhaust fan(s) complete with explosion-proof motor(s) that match 6 ACPH
- indoor force air heater(s), again with explosion-proof motor(s), to compensate conduction and leakage heat losses
- a fresh make-up air unit (6 ACPH supply, outlet 20°C) or a centralized supply
- louvre, registry, temperature control and purge cycle for shutdown and start-up.

Barrel Transfer Facilities

Feedstock which is generally shipped in large drums or standard oil barrels will require:

- a temporary storage and loading/unloading pad and
- a transfer mechanism for emptying the content to Feed Tank (T-1).

III. CANADIAN HYDROTREATING TEST CENTRE (CHTC) (cont'd)

4. FACILITY DESIGN (cont'd)

An area in the Plot (the high Pressure Bay, drawing 140-B2001) is identified for storage and loading.

The most convenient transfer mechanism is an air-operated (pneumatic) Barrel Transfer Pump complete with flexible hose and quick connects/coupling. For the size of Feed Tank, the Barrel Transfer Pump would require only a rated flow of 5 US gallons per minute and a differential head of 30 ft., self-lubricating by air force and a strainer are also required.

Utilities and Miscellaneous

These include, as a minimum,

- a hydrocarbon vent header
- an oily water floor sump
- a portable ladder and a work/sample bench
- a hose station
- utilities outlets
 - o Instrument Air
 - o Plant Air
 - o Cooling Water (supply and return)
 - o Nitrogen and hydrogen cylinders enclosure
 - o Steam (15# Pressure sufficient)
 - o condensate return
 - o Hot Water (for washdown)

4.4 Downstream Processing Units

Contingent upon availability of plot area, it is recommended that a Stabilizer and a FCC unit be included as part of CHTC.

IV. CONCLUSIONS AND RECOMMENDATIONS

IV. CONCLUSIONS AND RECOMMENDATIONS

Referring to Section 1, Chapter I, we can draw the essential conclusion that the Objective set forth for this contract has been achieved. Based on the work and results as detailed in the previous chapters, including SRC's Subcontractor Report, several recommendations can also be made.

CONCLUSIONS

1. Canadian Synthetic Crude Distillates can be conventionally hydrotreated, using commercially available hydrotreating catalysts.

This has been substantiated by the results of screening and detailed hydrotreating tests, which indicated:

- a. excellent desulphurization (to 500 ppm or less) and denitrogenation (basic nitrogen reduced to 100 ppm or less in most cases) were achieved,
 - b. very minor catalyst deactivation was observed,
 - c. good quality hydrotreated liquid products suitable for FCC feed were produced, and
 - d. for Suncor Coker Gas Oil, Shell S-424 was assessed as the best catalyst.
2. Canadian hydrotreating test expertise has been proven and enhanced.

Findings and results derived from the contract work will prove valuable for continuing technology development and as well, form data/information basis for immediate expansion of hydrotreating R & D projects.

3. Based on test results on the bench-scale hydrotreating unit, a down-sized demonstration plant entitled "Canadian Hydrotreating Test Centre (CHTC)" has been designed. This consists of:
 - a. Process design and material balance using bench scale test parameters.
 - b. Piping and instrumentation design complete with major equipment sizing (Appendix I Equipment List) and,

IV. CONCLUSIONS AND RECOMMENDATIONS (cont'd)

CONCLUSIONS (cont'd)

- c. Preliminary plot layout plan using High Pressure Laboratory at SRC as the plant site.

During the course of experimental work, potential areas of improvement and deficiency were discovered. These have been addressed in several places in Subcontractor Report and are collated below as recommendations.

RECOMMENDATIONS

1. The following should be implemented to improve hydrotreating test, for future work:
 - a. extended on-stream test period of three months or longer is required to sensibly assess catalyst aging characteristics,
 - b. experimental system capability, in terms of reactor size or feed rate or LHSV, should be increased in order that adequate products can be collected for extensive analytic uses, and
 - c. further test work with wider range of process parameters (Pressure, Temperature, LHSV and Hydrogen Rate) is needed to determine the optimal hydrotreating condition (the current test condition was felt on the higher severity side than actually required).
2. It is strongly recommended that a Canadian Hydrotreating Test Centre be built, fulfilling the long term objective of this technology development project. The work would involve:
 - a. detailed design and cost estimate for a CHTC based on what is presented in Chapter III,
 - b. liaison and discussion with potential users from industry and R & D organization to firm up the scope and funding arrangement, and
 - c. procurement and construction of the plant.

IV. CONCLUSIONS AND RECOMMENDATIONS (cont'd)

RECOMMENDATIONS (cont'd)

We would further recommend that Petroleum Division, Saskatchewan Research Council be the selected site with some custodian rights.

The recommended work in 1. above would indeed be carried out using the CHTC facilities. In addition, testing of other feedstocks, such as distillates from Syncrude Canada, and other catalysts, such as the newly marketed, pre-sulphided Eurecat hydrotreating catalysts can all be done in one, single complete system.

APPENDICES

- A. CATALYSTS DATA FROM SUPPLIERS
- B. HYDROTREATING SCREENING TESTS DATA AND MATERIAL BALANCE
- C. PROCESS FLOW DIAGRAM (PFD) - CANADIAN HYDROTREATING TEST CENTER, DRAWING NO. 080-B3000
- D. PIPING AND INSTRUMENTATION DIAGRAM (P & ID) - CANADIAN HYDROTREATING TEST CENTRE, DRAWING NO. 080-D3001
- E. PLOT PLAN - CANADIAN HYDROTREATING TEST CENTRE, DRAWING NO. 140-B2001
- F. EQUIPMENT LIST - CANADIAN HYDROTREATING TEST CENTRE
- G. PROJECT STAFF LIST

A. CATALYSTS DATA FROM SUPPLIERS

- o KETJEN KF-840
- o HALDOR-TOPSOE TK-581
- o CYANAMID HDN-30
- o CYANAMID HDS-20
- o SHELL S-424 (NOT AVAILABLE FROM SHELL)

Hydroprocessing Catalysts TK-551/TK-581

The TK-series of hydroprocessing catalysts have, since 1979, been successfully used in many, widely differing, hydroprocessing applications from naphtha through lube-oil and vacuum resid.

The TK-catalysts are available in different types which can conveniently be divided into three specialized application areas

- 1) Hydroprocessing of VGO and VGO/RESID blends prior to fluid catalytic cracking.
- 2) Hydroprocessing of resids using the concept of "Composite Catalyst Fillings".
- 3) Hydroprocessing of all refinery cuts using active Raschig Ring type catalysts to alleviate pressure drop problems.

The extensive R&D program behind the TK-catalysts has led to a thorough understanding of the fundamentals of hydroprocessing. It has become clear that selection of catalyst is not straightforward as the optimum choice depends strongly on feedstock type, operating conditions and product requirements. Often the best choice is not a single catalyst but a combination of several types in a "Composite Catalyst Filling".

The fact that all TK-catalysts are available in a variety of sizes, and also in the form of Raschig Rings, gives greater flexibility in reactor loadings and in grading of catalyst beds not only for activity but also for pressure drop.

The TK-catalysts are suitable for both normal and dense loading such as the ARCO licensed Catalyst Orientated Packing (COP) method. As with other catalyst types about 15% more TK catalyst can be loaded when using this technique.

Our specialists – with the full backing of our advanced R&D facilities – are available to assist customers in making the right choice of catalysts.

Principal Functions

TK-551/581 are Ni/Mo type catalysts for heavy atmospheric and vacuum distillates where they accomplish:

- Nitrogen removal to low levels
- Saturation of polynuclear aromatics
- Saturation of olefins
- Deep desulfurization

Application Areas

- Pretreatment of Fluid Catalytic Cracking feedstocks.
- Treatment of atmospheric and vacuum gas oils.
- Deep HDS of demetallised resids.
- Hydroprocessing of de-asphalted oil (D.A.O.)

Main Features

- High HDN activity
- High HDS activity
- High activity for PNA saturation
- High hydrogenation activity

Packing & Storage

All TK-catalyst types are supplied in easy to handle fibre drums of 3.9 cuft capacity (110 liters) with a removable lid. Alternatively the catalysts can be supplied in steel drums of the same size. For larger quantities catalysts can be supplied in "Big Bags" holding 35 cuft (1 cubic meter). The big bags are equipped with a bottom discharge chute.

In all cases an internal polyethylene liner protects the catalysts from ingress of water or moisture. It is advisable to store catalysts indoors, but if for some reason they must be stored outside, the catalyst containers must be placed on pallets or logs and be securely covered with a plastic or canvas sheet to protect them from rain.

Typical Properties

	TK-551		TK-581	
	Rings	Cylinders	Rings	Cylinders
NiO, % wt	3.4	3.4	3.4	3.4
MoO ₃ , % wt	14	14	14	14
Bulk density, lbs/cuft kg/l	37 0.60	46 0.74	37 0.60	46 0.74
Attrition loss ¹⁾ , % wt	<2	<0.2	<2	<0.2
Surface area, m ² /g	180	180	140	140
Pore volume, cc/g	0.45	0.45	0.45	0.45
Side crush strength ¹⁾ , lbs/mm kg/mm	1.6 0.7	4.4 ²⁾ 2.0	1.3 0.6	3.5 ²⁾ 1.6

Sizes:

Rings: 3/16" ODx3/32" ID

Cylinders: 1/32", 1/20", 1/16"

1) Topsøe method available on request. 2) 1/16" cylindrical extrudates.

Description

Both TK-551 and TK-581 are Ni/Mo catalysts but differ in the structure of the catalyst carrier. TK-581 has wider pores, and is therefore recommended for heavy feeds, where pore diffusion is a restriction. TK-551 is the more active catalyst for lighter feedstocks. The difference between the two catalysts is illustrated by the examples in Table 1.

Both types combine high HDN activity and high hydrogenation activity with very high activity for HDS. TK-581 is well suited for the heaviest VGO's and DAO's while TK-551 is recommended for atmospheric gas oils and lighter VGO. Because of their excellent hydrogenation activity both types are particularly suited for FCC pretreatment and for cracked feedstocks. Some examples of the use of TK-551 and TK-581 are shown in Table 2.

Table 1.

Feedstock	North Slope/Calif. VGO		Mixed Coker Gas Oil HGO, VGO	
°API	20.1		26.3	
Distill., °F 10/50/90 vol %	662/837/939		459/662/865	
S, % wt	1.23		0.99	
N, ppm wt	2580		2390	
Conradson Carbon, wt %	0.49		0.16	
Refr. Index	1.52135		1.49865	
Operation				
LHSV hr ⁻¹	1.5		1.5	
H ₂ pressure psig kg/cm ² g	1400 98		850 60	
Temperature, °F °C	705 374		700 371	
Catalyst	TK-551	TK-581	TK-551	TK-581
Product				
°API	24.1	24.3	29.8	29.6
S, % wt	0.09	0.06	0.04	0.08
N, ppm wt	1170	990	1200	1400
Conradson Carbon, wt%	0.05	0.04	0.03	0.03
Refr. Index	1.50410	1.50270	1.48660	1.48670

Table 2.

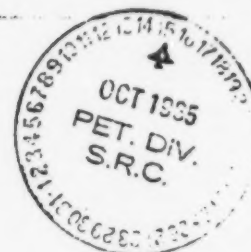
Catalyst Type	TK-551		TK-581			
Feedstock	LGO		VGO		40% VGO 60% Coker GO	
LHSV hr ⁻¹	3.5		2.0		1.35	
H ₂ pressure psig kg/cm ² g	710 50		925 65		640 45	
Temperature, °F °C	626 330		698 370		732 389	
	Feed	Prod.	Feed	Prod.	Feed	Prod.
°API	38.0	43.2	21.3	27.6	21.2	26.8
S, % wt	1.09	0.10	2.41	0.17	2.59	0.09
N, ppm wt	44	15	880	330	2200	1050
C/H Ratio, wt/wt	6.48	6.36	7.07	6.84	7.26	6.94
CCR, % wt	-	-	0.76	0.07	0.40	0.05
Brom. No.	-	-	4.7	1.0	17.5	1.2
H ₂ consumption; SCF/BBL	150		350		450	

CATALYST TEST CERTIFICATE

18-03

KETJENFINE®

Hydrotreating Catalyst

Grade: KF-840-1.30

Test no. : R-507
 Shipment to : Saskatchewan Research Council
 Order # : APL-85-31
 Lot # :
 Shipment date : 1 October 1985

Packing : Plastic Poly Bottle
 Quantity : 1 Liter
 Transportation : U.S. Mail

CHEMICAL ANALYSIS

Loss on ignition (at 550°C)	: 1.2	% wb *
MoO ₃	: 18.8	% db *
CoO	: -	% db
NiO	: 4.0	% db
Na ₂ O	: .06	% db
SO ₄	: 1.2	% db
Fe	: .01	% db
SiO ₂	: .52	% db

PHYSICAL PROPERTIES

Surface Area	: 172	m ² /g
Pore Volume (H ₂ O)	: .414	ml/g
Estimated reactor density	: .70	g/ml
Compacted bulk density	: -	g/ml
Av. length	: 3.1	mm
Av. diameter	: 1.14x1.33	mm
Abrasion	: .43	%
Av. side crushing strength	: 7.7	lbs/mm

* wb = wet basis db = dry basis

Martin Whelan
 Manager, Analytical Services

AERO^{*} TRILOBE^{*} HDS-20

HYDROTREATING CATALYST
is the most active
hydrodesulfurization
catalyst commercially
available.

Summary:

More efficient use of active materials, more efficient than conventional catalysts.

Improved pore size distribution, particle size, composition and chemistry of the metals — cobalt molybdenum on alumina — all produce superior activity.

Unique, more effective catalyst size and shape provide activity advantages of smaller catalyst particle without sacrificing pressure drop; increased catalyst wetting because of the great surface to volume ratio.

Improved oil distribution reduces length of diffusional path within the catalyst particle, improving utilization of catalyst properties.

Maximum desulfurization solves many problems related to stringent sulfur emission regulations and high fuel costs.

Designed for mixed phase hydrotreating of middle distillate stocks, it also excels in treating heavier feeds and naphthas.

Excellent nitrogen removal capability for a cobalt molybdenum catalyst.

Superior activity permits

- Increased throughput
- Reduced fuel costs
- Lower operating temperatures, which in turn mean lower corrosion rates
- Longer catalyst life
- Lower catalyst costs
- Lower capital investment in new reactors

Two sizes designed — for pressure drop equivalence to 1/8-inch cylindrical extrudates, use HDS-20; — for pressure drop equivalence to 1/16-inch cylindrical extrudates, use HDS-20A.

Advantages: AERO TRILOBE HDS-20 Catalyst

Maximum desulfurization activity

AERO TRILOBE HDS-20 catalyst combines optimized particle shape, pore size distribution and composition for outstanding desulfurization activity in commercial mixed phase hydrotreaters. Its unique design uses active metals more efficiently than conventional catalysts due to its patented shape and optimized pore size distribution. Pilot plant data show AERO TRILOBE HDS-20 catalyst to be the most active hydrodesulfurization catalyst commercially available.

High denitrogenation activity

AERO TRILOBE HDS-20 catalyst has remarkable denitrogenation activity for a cobalt molybdenum catalyst. This feature makes AERO TRILOBE HDS-20 catalyst a particularly versatile performer. In applications where nitrogen as well as sulfur removal is needed, but nickel molybdenum catalysts cannot be justified, AERO TRILOBE HDS-20 catalyst will excel.

High void fraction

Because of the unique shape of AERO TRILOBE HDS-20 catalyst, it packs with more free space (void fraction) between catalyst particles than conventional spherical or cylindrical catalysts. Consequently there is more void volume in the reactor to accumulate scale and other debris. Higher void fractions make pressure drop less sensitive to scale accumulation. This feature means that AERO TRILOBE HDS-20 catalyst has the potential for extended run life between shutdowns caused by pressure drop problems.

Low pressure drop

The unique shape of AERO TRILOBE HDS-20 catalyst and the resulting high void fraction permit the use of effectively smaller catalyst particles without a corresponding increase in pressure drop. Consequently, the activity advantage of smaller catalyst particles has been achieved without the disadvantages of higher pressure drops.

Greater external surface area vs cylinders or spheres

On an equivalent pressure drop basis, AERO TRILOBE catalyst has about 60% more external surface area than spheres and almost 50% more than cylindrical extrudates. Even on an equal particle diameter basis, where cylinders and especially spheres have higher pressure drop, TRILOBE catalyst still has more external surface area. With a much lower pressure drop, HDS-20 has in addition 20% more external surface area per bulk volume than does a $\frac{1}{8}$ " sphere. This could result in greater metals removal and better tolerance to contaminants deposition.

Improved oil distribution

AERO TRILOBE HDS-20 catalyst, unlike conventional catalysts, is designed to improve liquid distribution (increase wetting) in mixed phase hydrotreating reactors. This is accomplished by its unique shape which permits a relatively high surface to volume ratio and the "troughs" contained in each catalyst particle. The troughs are formed by the three lobes of the catalyst. In essence, each catalyst particle acts as a miniature oil distributor by continuously changing direction of the oil. The net result is increased oil holdup in the reactor resulting in more efficient use of the catalyst. This unusual oil distribution feature should prove advantageous in all commercial reactors, particularly those with oil distribution problems.

High crush strength

AERO TRILOBE HDS-20 catalyst is mechanically designed for high crush strength. Each lobe is an integral part of the structure and will not break off in normal use. Typical crush strengths for AERO TRILOBE HDS-20 and HDS-20A catalysts are 21 lb (9.5 kg) and 16 lb (7.3 kg), respectively.

Low density

AERO TRILOBE HDS-20 catalyst has a relatively low density for a high performance catalyst, typically 41 lb/ft³ (657 kg/m³) commercially loaded using a standard hopper and sock loading technique. Some catalysts attempt to achieve high volume activity by maximizing density. High density catalysts are costly because more pounds are required to fill a reactor. The relatively low density but high volume activity of the AERO TRILOBE HDS-20 catalyst makes it most economical on a cost-performance basis.

Table I

TEST FEEDSTOCK DESCRIPTIONS			
	Furnace Oil*	VGO-A	VGO-B
Gravity, °API	28.8	21.4	19.9
Sulfur, wt %	1.6	2.4	2.5
Total Nitrogen, wt ppm	265	970	1,120
ASTM Distillation			
IBP	386°F (197°C)	707°F (375°C)	672°F (356°C)
10%	440°F (227°C)	799°F (426°C)	796°F (424°C)
50%	514°F (268°C)	843°F (451°C)	890°F (477°C)
90%	620°F (327°C)	905°F (485°C)	1,032°F (556°C)
E.P.	688°F (364°C)	960°F (516°C)	—
FIA, vol %			
Aromatics	50	—	—
Olefins	3	—	—
Saturates	47	—	—
	100		
Pour Point, ASTM			
	—	100°F (38°C)	110°F (43°C)
Conradson Carbon, wt %			
	—	0.9	1.2
*Blend: 50% light catalytic cycle oil—50% straight run distillate			

Performance: Furnace Oil Tests

Superior desulfurization with less catalyst

AERO TRILOBE HDS-20A catalyst's superior desulfurization capability was demonstrated on a typical furnace oil. A description of this feedstock is given in Table I. Tests were conducted at 500 psig, 500 SCF/bbl H_2 , temperatures from 600°-700°F (315°-370°C), and volume space velocities ranging from 3-8. Results at 650°F (345°C), Figure 1, show AERO TRILOBE HDS-20A catalyst to be markedly more active than AERO HDS-2A and AERO HDS-16A catalysts. For example, AERO TRILOBE HDS-20A catalyst achieves 85% desulfurization with less than half the amount of catalyst required for AERO HDS-2A catalyst (3.5 vs 8 LHSV).

Unique design — surpasses other high performance catalysts

Sulfur removal data shown in Figure 1 are quantized in terms of relative weight and volume activities in Table II. Relative activity is defined here as the ratio of second order rate constants with AERO HDS-2A catalyst chosen as the reference point with assigned activities of 100. At these conditions AERO TRILOBE HDS-20A catalyst was 65% more active on a weight basis and 120% more active on a volume basis than AERO HDS-2A catalyst. Its unique design puts it in a league by itself, setting the pace for high performance catalysts.

Exceptional increasing temperature response

Sulfur removal as a function of process temperature is displayed in Figure 2. These data show an unusual feature of AERO TRILOBE HDS-20 catalyst — an increase in relative activity with increasing temperature. For example, at 75% desulfurization AERO TRILOBE HDS-20A catalyst is 30°F (17°C) more active than AERO HDS-2A

catalyst, but at 90% desulfurization the activity advantage increases to over 40°F (22°C). This unusual high temperature response is the result of HDS-20's higher effectiveness factor which permits very efficient use of active metals. This will generally be seen in applications where diffusion resistance is moderate.

Higher activation energy

Sulfur removal data as a function of temperature, shown in Figure 2, are compared in terms of relative volume activity in Table III. Relative activities are calculated as the ratio of second order rate constants and are referenced to AERO HDS-2A catalyst with an assigned activity of 100 at each temperature. These data show the relative activity of HDS-16A to be invariant with process temperature. AERO TRILOBE HDS-20A catalyst excels at all process temperatures. Its exceptional high temperature response property is manifested by its increase in relative activity with increasing temperature (higher activation energy). This is exemplified by noting AERO TRILOBE HDS-20A catalyst increases in relative activity from 190 to 220% as the temperature is increased from 600° to 650°F (315°-345°C).

High denitrogenation for a CoMo catalyst

AERO TRILOBE HDS-20A catalyst also provides high denitrogenation capability for a cobalt molybdenum catalyst. Total nitrogen removal from the furnace oil was measured at 650°F (345°C), 500 psig, 500 SCF/bbl H_2 and volume space velocities ranging from 3-8.

Data in Figure 3 show AERO TRILOBE HDS-20A catalyst to have more than twice the nitrogen removal activity of AERO HDS-2A catalyst. At 3 space velocity it removes 65% of the total nitrogen whereas HDS-2A removes less than 40%.

Figure 1

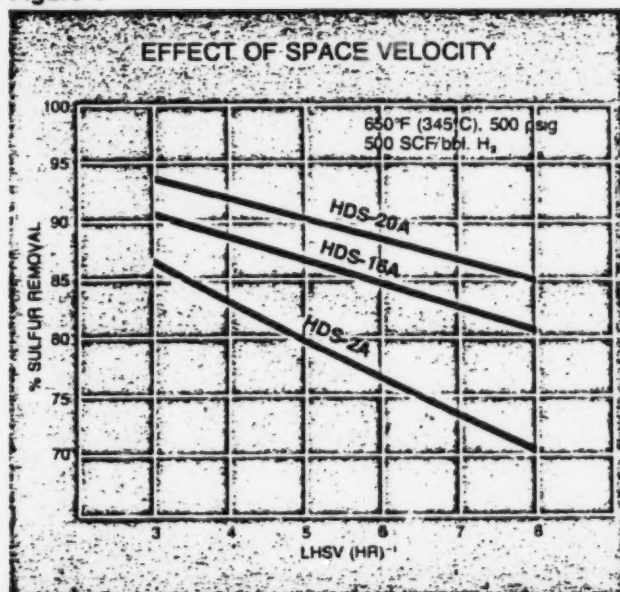


Table II

Catalyst	Sulfur Removal Activity Comparison	
	Relative Activities*	
	Weight Basis	Volume Basis
HDS-2A	100	100
HDS-16A	110	165
AERO TRILOBE HDS-20A catalyst	165	220

*Second order kinetics

Table III

Catalyst	Sulfur Removal Activity vs Temperature		
	Relative Volume Activity		
	600°F (315°C)	650°F (345°C)	700°F (370°C)
HDS-2A	100	100	100
HDS-16A	165	165	165
AERO TRILOBE HDS-20A catalyst	190	220	250

*Second order kinetics

Figure 2

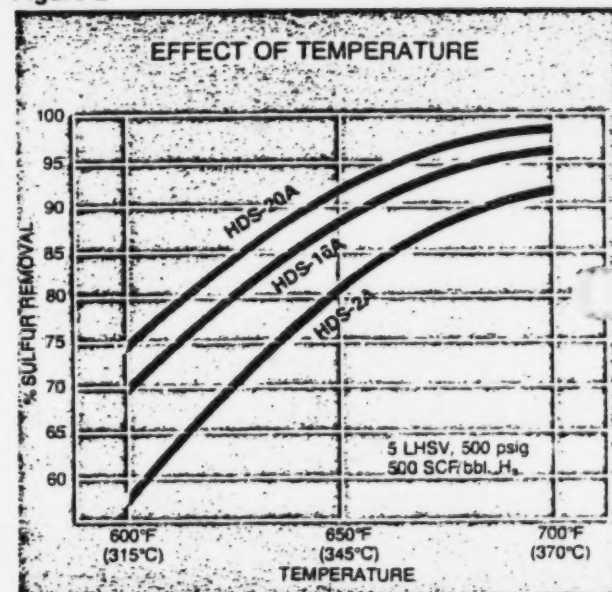
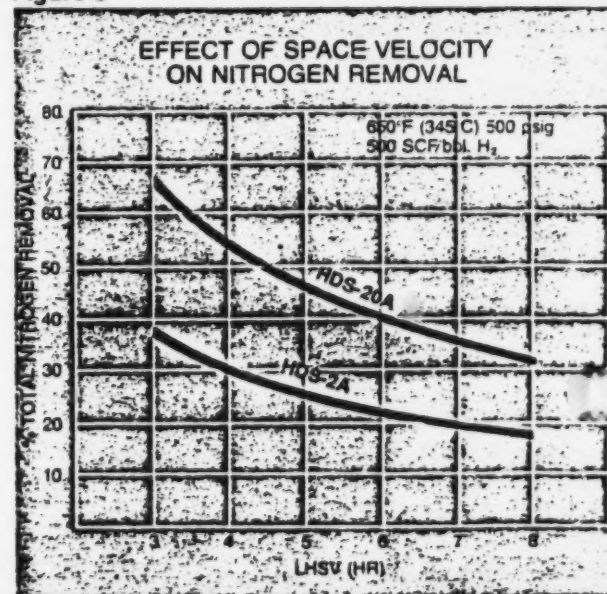


Figure 3



Performance:

Vacuum Gas Oil Tests

AERO TRILOBE HDS-20A catalyst's superior ability to remove sulfur from vacuum gas oils was demonstrated on two different feedstocks. Inspections of these vacuum gas oils are given in Table II. Tests were conducted at 1,000 psig, 1,500 SCF/bbl H_2 , temperatures from 650°-750°F (345°-400°C) and space velocities from 1-3.

70% greater space velocity

Sulfur removal as a function of space velocity for VGO-A is shown in Figure 4. AERO HDS-1441A, a Cyanamid catalyst with commercially proven performance in treating VGO, was chosen as the reference catalyst. In this case AERO TRILOBE HDS-20A catalyst is markedly superior to AERO HDS-1441A catalyst and competitive Catalyst Z. At 85% desulfurization, space velocities of the order of 1.5 are required for HDS-1441A and Catalyst Z whereas HDS-20A accomplishes the same sulfur removal at a space velocity greater than 2.5.

Desulfurization advantage >35°F (20°C)

Figure 5 compares sulfur removal in vacuum gas oil A as a function of temperature for these catalysts. Again, AERO TRILOBE HDS-20A catalyst is shown to be substantially more active than HDS-1441A and competitive Catalyst Z. For example, at 85% desulfurization HDS-20A has an activity advantage of 35°-40°F (20°-22°C).

Substantial activity advantages

Data shown in Figure 4 for the desulfurization of vacuum gas oil A have been summarized in terms of relative volume activity in Table IV. HDS-1441A was chosen as the reference with an assigned activity of 100. Relative activities were computed as the ratio of 3/2 order rate constants. This analysis shows that AERO TRILOBE HDS-20A catalyst is 90% more active than HDS-1441A while Catalyst Z is only 15% more active.

Desulfurization of a different vacuum gas oil, VGO-B, as a function of space velocity is shown in Figure 6. For this oil the magnitude of AERO TRILOBE HDS-20A catalyst's activity advantage is less dramatic than when treating VGO-A but nevertheless substantial. Data analyses summarized in Table IV show AERO TRILOBE HDS-20A catalyst to be 45% more active than HDS-1441A. Data for another competitive catalyst, Catalyst X, show it also to be inferior to HDS-20A.

Superior desulfurization generates economical benefits

AERO TRILOBE HDS-20A catalyst has been shown to possess superior desulfurization activity in furnace and vacuum gas oil tests. Its higher activity will prove advantageous in increasing throughput, reducing fuel costs, increasing catalyst life, lowering catalyst costs, and decreasing capital investment in new reactors.

Figure 4

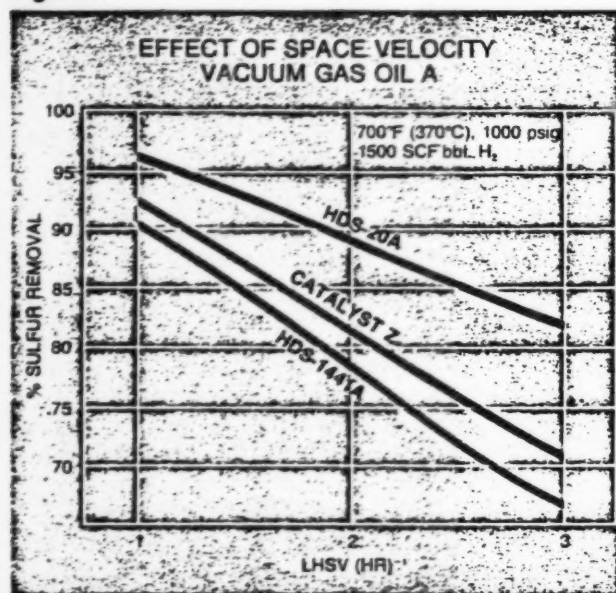


Figure 5

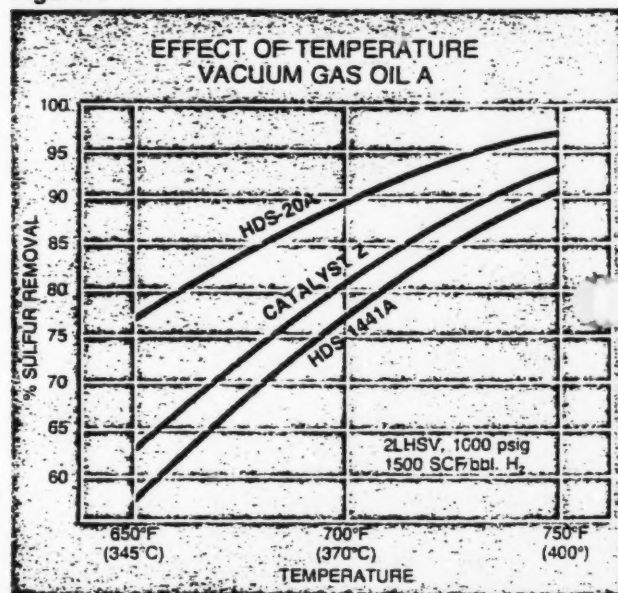


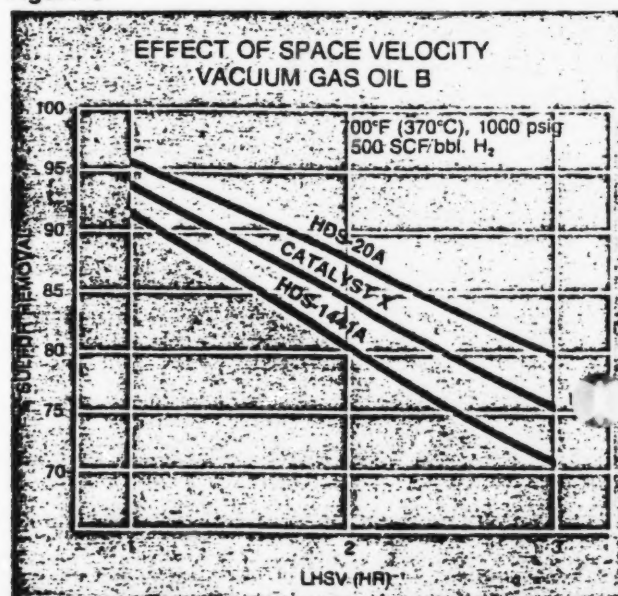
Table IV

Desulfurization Activity Comparison

Catalyst	Relative Volume Activity*	
	VGO-A	VGO-B
HDS-1441A	100	100
AERO TRILOBE		
HDS-20A catalyst	190	145
Catalyst X		125
Catalyst Z	115	

Based on 3/2 order kinetics

Figure 6



Metals removal

In addition to promoting the various hydrogen treating reactions discussed previously, AERO TRILOBE HDS-20 catalyst removes metals from refinery feedstocks. This benefits the refiner by protecting downstream operation. For example, the pretreater before a naphtha reformer protects the extremely valuable reforming catalyst that can be readily poisoned by relatively small amounts of metals.

Metals removal generally is desirable, but these metals are hydrogen treating catalyst contaminants, and in sufficient concentrations will decrease activity. The metals of concern are sodium, iron, lead, arsenic, zinc, copper, nickel and vanadium — the first four being the most common. Contamination may also be caused by excessive amounts of sulfates, carbon, or silica.

Some comments on the most common metal contaminants are given in this section.

Sodium – There appears to be no data relating catalyst activity solely to sodium level. It is believed that sodium indirectly acts by promoting sintering similar to the manner in which it acts on a fluid cracking catalyst. The physical problem of simple pore filling due to salt deposition also is suspected.

Arsenic – Analyses at various bed levels indicate that arsenic tends to deposit on the catalyst in layers. Each layer retains arsenic fairly well, up to a saturation point, at which time breakthrough to the next layer occurs. The catalyst must be replaced when arsenic breaks through the final layer into the exit stream in appreciable quantity.

Arsenic is thought to be a more serious poison to hydrogen treating catalyst than lead, but the breakthrough and subsequent poisoning of downstream catalysts still is the major problem.

Lead – Lead and arsenic depositions differ in that lead apparently distributes itself more evenly throughout the catalyst bed, rather than forming layers as does arsenic. As with arsenic, breakthrough is the main problem, and its fairly uniform distribution in the bed can lead to a quicker breakthrough. Lead removal is maximized by the proper relationship of pore size to surface area. Lead laydown is felt to be irreversible to regeneration. Metallic lead, lead sulfides or lead oxides are believed to be formed and remain on the catalyst.

Iron – Little specific information on the catalytic effects of iron contamination is available. However, it undoubtedly contributes to the overall metals saturation level of HDS catalysts. If nothing else, iron increases the level of particulate matter in the bed and causes pressure drop.

Typical Properties

Table V

CHEMICAL (wt %, dry basis)		
MoO ₃		16.2
CoO		5.0
Na ₂ O		0.05
SO ₄		0.3
PHYSICAL		
Poured Bulk Density	 41 lb/ft ³ (657 kg/m ³)	
Compacted Bulk Density	 46 lb/ft ³ (737 kg/m ³)	
Surface Area, m ² /g		230
Pore Volume (H ₂ O), cc/g		0.52
	HDS-20A	HDS-20
Average Diameter*	0.051 in. (0.13 cm)	0.100 in. (0.25 cm)
Average Length	0.16 in. (0.41 cm)	0.20 in. (0.51 cm)
Average Crush Strength	16 lb. (7.3 kg)	21 lb. (9.5 kg)
Loss on Abrasion, wt %	0.5	1.3

*Diameter is measured across two lobes and is slightly less than the diameter of the circumscribed circle.

Precautions

AERO TRILOBE HDS-20 catalyst contains insoluble cobalt compounds and insoluble molybdenum compounds. Exposure to such compounds as airborne particulates must be limited according to the provisions of Section 1910.1000 of the regulations under the U.S. Occupational Safety and Health Act (29 CFR 1910.1000).

Important Notice

The information and statements herein are believed to be reliable, but are not to be construed as a warranty or representation for which we assume legal responsibility. Users should undertake sufficient verification and testing to determine the suitability for their own particular purpose or information or products referred to herein. NO WARRANTY, FITNESS FOR A PARTICULAR PURPOSE IS MADE. Nothing herein is to be taken as permission, inducement or recommendation to practice any patented invention without a license.



TRILOBE HDN-30

The N stands for Nitrogen.

TRILOBE HDN-30 catalyst is the most active hydrodenitrogenation catalyst commercially available.

Unique, more effective catalyst size and shape provide activity advantages of smaller catalyst particle without increasing pressure drop.

Improved oil distribution is accomplished by unique catalyst shape which results in high external surface area to volume ratio and "troughs" in each catalyst particle. Each acts as a miniature oil distributor by continuously changing the direction of oil flow. The net result is increased oil holdup in the reactor, resulting in more efficient use of the catalyst. This unusual oil distribution feature should prove advantageous in all commercial reactors.

Two sizes designed - TRILOBE HDN-30 (3.2) catalyst for pressure drop equivalence to 3.2 mm ($\frac{1}{8}$ inch) diameter cylindrical extrudates.

TRILOBE HDN-30 (1.6) catalyst for pressure drop equivalence to 1.6 mm ($\frac{1}{16}$ inch) diameter cylindrical extrudates.

High crush strength - TRILOBE HDN-30 catalyst is designed for high crush strength. Each lobe is an integral part of the structure and will not break off in normal use. Typical crush strengths for TRILOBE HDN-30 catalyst are 5 lb/mm (2.3 kg/mm) for type 3.2 and 4 lb/mm (1.8 kg/mm) for type 1.6.

Increased metals removal - On an equivalent pressure drop basis, TRILOBE catalyst has about 50% more external surface area than cylindrical extrudates. This extra surface area, combined with a very high level of active metal, results in both substantially increased metals removal and greater tolerance to contaminants deposition on the catalyst.

Greatly improved efficiency factor - TRILOBE HDN-30 (3.2) catalyst combines the benefits of "large" diameter catalysts - decreased initial pressure drop and decreased pressure drop caused by fouling of the catalyst bed - with catalytic activity far superior to any conventionally shaped 3.2 mm ($\frac{1}{8}$ inch) NiMo catalyst.

Designed for hydrotreating of vacuum gas oils, lube oils, coker naphthas and other feeds - wherever maximum obtainable denitrogenation combined with excellent sulfur removal is needed. The catalyst enhances cat cracker performance, improves color in lube stocks, permits increased unit thruput in capacity-limited units, allows lower fuel costs by reducing processing temperatures.

Moderate density - TRILOBE HDN-30 catalyst has a moderate density for a new generation, high metals denitrogenation catalyst, typically 46 lb/ft³, (735 kg/m³), commercially loaded using standard hopper and sock loading techniques.

Where the maximum obtainable denitrogenation activity of TRILOBE HDN-30 is not required, see your Cyanamid Technical Representative about TRILOBE HDS-3. This hydrogen treating catalyst combines the many advantages of Cyanamid's shaped catalyst technology into a relatively low density nickel-molybdenum catalyst with enhanced denitrogenation and desulfurization activity and very economical cost per fill.

Pilot Plant Tests

Maximum obtainable denitrogenation

The superior denitrogenation capabilities of TRILOBE HDN-30 catalyst have been demonstrated on several typical feedstocks.

Nitrogen removal results (Figure 1) show TRILOBE HDN-30 (1.6) catalyst to be substantially more active than conventionally shaped HDS-3 (1.6). It is superior to competitive high density high metals nickel-molybdenum catalysts.

See Table II for a description of Gas Oil A. Tests were conducted at 800 psig (55 bar), 2000 SCF/bbl (356 normal m³/m³) H₂, 2.5 LHSV, temperatures from 600°-700°F (315°-370°C).

Nitrogen removal data from Figure 1 are shown in Table I. The volume activity of TRILOBE HDN-30 (1.6) catalyst averages 30% higher than HDS-3 (1.6). Of particular interest are the weight activities of the catalysts. Manufacturers of nickel-molybdenum catalysts have increased catalyst metals loadings (and bulk densities) in an effort to increase activity. In some cases this has resulted in slightly higher volume denitrogenation activities. Unfortunately the weight activity is often lower than before.

Tyranamid's shaped catalyst technology has produced TRILOBE HDN-30, a catalyst that makes more efficient use of active metals and thus is more active on both a volume *and* weight basis.

Excellent desulfurization

Figure 2 illustrates the excellent sulfur removal characteristics of TRILOBE HDN-30 (1.6) catalyst. The basis for comparison, HDS-3 (1.6), is known throughout the world to be an excellent nickel-moly catalyst for both desulfurization and denitrogenation.

Many competitive high density, high metals nickel-molybdenum catalysts sacrifice sulfur removal efficiency to obtain improved nitrogen removal. With its unique patented shape, there is no desulfurization activity penalty with TRILOBE HDN-30.

Figure 1

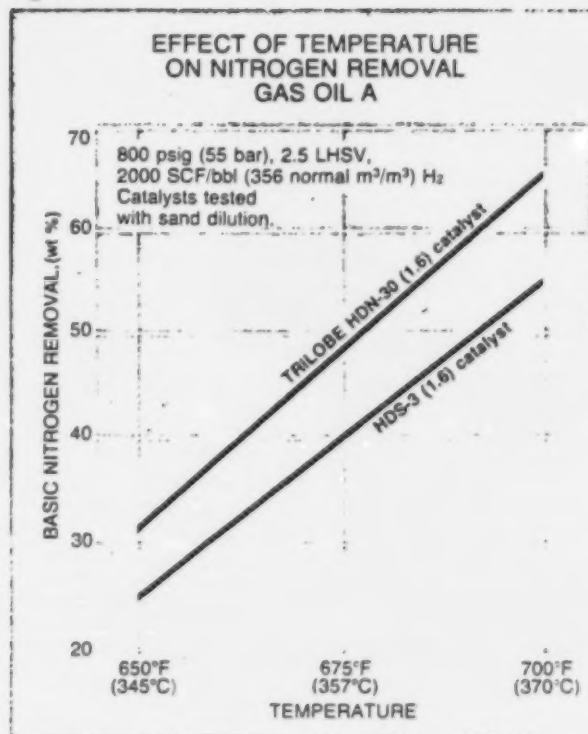


Table I (refers to Figure 1)

Nitrogen Removal Activity Comparison				
	Relative Activities*			
	Volume Basis		Weight Basis	
	650°F (345°C)	700°F (370°C)	650°F (345°C)	700°F (370°C)
HDS-3 (1.6) catalyst	100	100	100	100
TRILOBE HDN-30 (1.6) catalyst	127	133	112	118

*First order kinetics

Figure 2

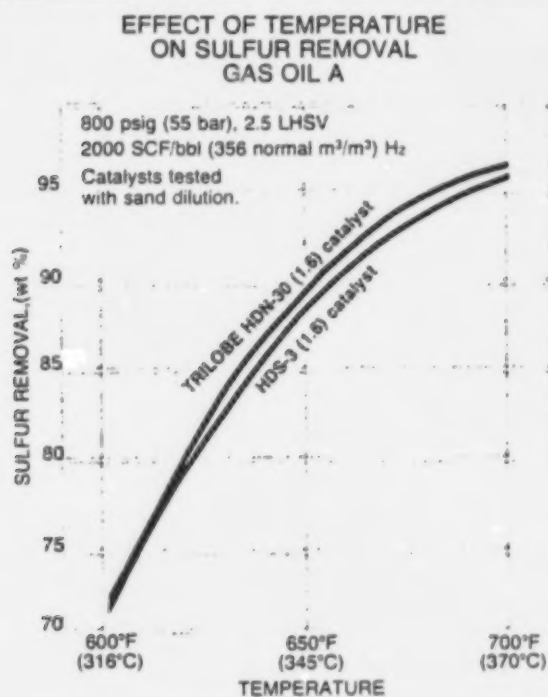


Figure 3

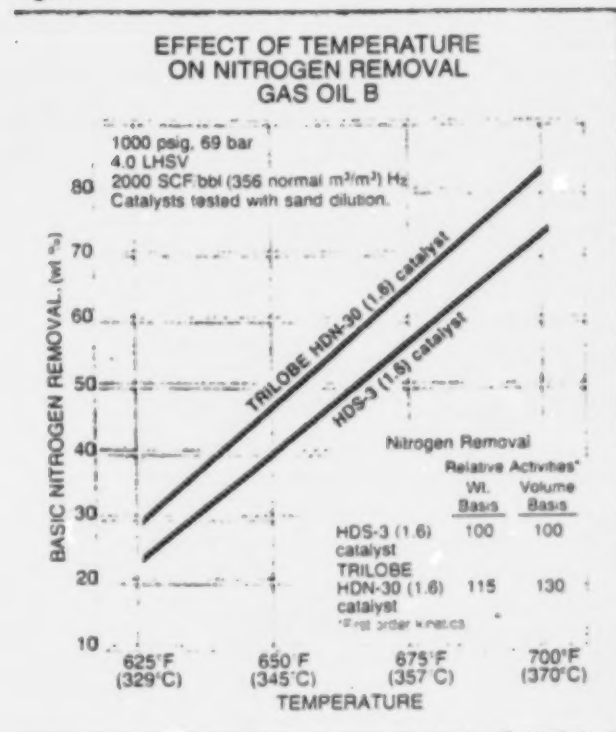


Table II

Test Feed Stock Descriptions				
Description	Gas Oil A		Gas Oil B	
	50/50 blend of Vacuum Gas Oil and FCC Light Cycle Oil		50/50 blend of West Coast Light Vacuum Gas Oil and FCC Light Cycle Oil	
Gravity, °API	24.6		23.6	
Basic Nitrogen, ppm	710		284	
Total Nitrogen, ppm	1915		1455	
Sulfur, wt %	1.35		1.40	
ASTM Distillation				
	°F	°C	°F	°C
IBP	250	121	463	239
10%	512	267	540	282
50%	626	330	635	335
90%	767	408	789	429
95%	837	447	—	—
EP	—	—	870	466

Denitrogenation activity confirmed

Testwork performed on Gas Oil B (Figure 3) confirms the superior denitrogenation activity of TRILOBE HDN-30 (1.6) catalyst versus HDS-3 (1.6). Tests were conducted at 1000 psig (69 bar), 4.0 LHSV, 2000 SCF/bbl (356 normal m³/m³) H₂ and temperatures 625°F to 700°F (329°C to 370°C). See Table II for feed stock description.

Again the volume activity of TRILOBE HDN-30 (1.6) catalyst averages 30% higher for denitrogenation than that of HDS-3 (1.6).

Typical Properties

Table III

CHEMICAL (wt %, dry basis)		
MoO ₃	20.5	
NiO	5.0	
Na ₂ O	0.03	
-SO ₄	0.3	
PHYSICAL		
Poured bulk density	46 lb/ft ³ (735 kg/m ³)	
Compacted bulk density	52 lb/ft ³ (830 kg/m ³)	
Surface area	160 m ² /g	
Pore volume (H ₂ O), cc/g	0.44	
	TRILOBE HDN-30 (3.2) catalyst	TRILOBE HDN-30 (1.6) catalyst
Size (pressure drop equivalent)	1/8" 3.2 mm	1/16" 1.6 mm
Average diameter* ..	0.10" 2.5 mm	0.050" 1.3 mm
Average length	0.20" 5 mm	0.15" 4 mm
Average crush strength**	5 lb/mm 2.3 kg/mm	4 lb/mm 1.8 kg/mm
*Diameter is measured across two lobes		
**Determined by 1/8" anvil method		

Efficiency factor

The tests for this brochure were conducted using TRILOBE HDN-30 (1.6) catalyst, the 1/16 inch (1.6 mm) pressure drop equivalent size, as this is the size more commonly used in test equipment. Frequently, a refiner will actually be installing larger size 1/8" (3.2 mm) catalyst into a commercial unit.

With its unique shape TRILOBE HDN-30 (3.2) catalyst, the pressure drop equivalent of a 1/8 inch (3.2 mm) diameter catalyst, normally demonstrates *greatly* increased performance relative to any conventionally shaped 1/8 inch (3.2 mm) catalyst.

In the hydrotreating of most feeds, diffusional limitations are a major concern. Because TRILOBE catalysts have an optimized three lobe design, the diffusion path in even the large size is relatively short. What this means to the refiner is:

- If you are presently using a 1/8 inch (3.2 mm) conventionally shaped Ni-Mo catalyst, a switch to TRILOBE HDN-30 (3.2) catalyst can often greatly increase activity while keeping the pressure drop characteristics of a large diameter catalyst.
- If you are presently using a 1/16 inch (1.6 mm) conventionally shaped Ni-Mo catalyst and pressure drop is limiting your run length, a switch to TRILOBE HDN-30 (3.2) catalyst can usually improve unit operation while not substantially affecting unit activity.

See your Cyanamid Technical Sales Representative for further information on how TRILOBE brand catalyst can improve your unit performance.

Precautions

TRILOBE HDN-30 catalyst contains insoluble molybdenum compounds. Exposure to such compounds as airborne particulates must be limited according to the provisions of Section 1910.1000 of the regulations under the U.S. Occupational Safety and Health Act.

B. HYDROTREATING SCREENING TESTS DATA

- o GAS PRODUCTS ANALYSES
- o LIQUID PRODUCTS ANALYSES
- o MATERIAL BALANCE

NOTES FOR TABLE

FEED

Gas Oil LHSV: Experimentally measured actual value.

H₂ Feed Rate: - Calculated by $L \times 900 / (LHSV \times 22,400 \times 0.9614)$
L = Apparent LHSV set by instrument.
Gas Oil Sp. Gr. = 0.9614
- Unit gmol/g of gas oil feed.

H/T LIQUID PRODUCT

Yield: Weight % of total gas oil feed.

Sulfur (S): Weight % of liquid product.

Basic N: Basic nitrogen content in Weight ppm (WPPM)
of liquid product.

Total N (TN): Total nitrogen in Wt. % of liquid product.

GAS YIELD

Expressed as Wt. % of total gas oil feed.

H₂ CONSUMPTION

This is a measured value computed to gmol/g of gas oil feed and percentage consumed of the hydrogen feed.

N%: Percentage of H₂ consumed NORMALIZED, based on hydrogen feed of 0.0417917 (or 0.0418) gmol/g.

CATALYST: Shell S-424

TABLE 1:

HYDROTREATING SCREENING TESTS SUMMARY
STEARNS CATALYTIC/SRC Project 4046
DSS/CANMET File No.: 068Q.23440-4-9318

RUN NO.	H/T CONDITION		LHSV	FEED		SP GR	H/T LIQUID PRODUCT		TN	GAS YIELD	H ₂ CONSUMPTION	
	P MPa	T °C	-	H ₂ gmol/g	-		YIELD Wt %	S Wt %	Wt %	Wt %	gmol/g	N %
A-1	10.5	360	1.06	0.0394	.8941	97.2	.305	50	.18	4.3	.0114	28.9
A-2	10.5	370	0.87	0.0480	.8912	96.0	.211	72	.11	3.1	.0299	62.3
A-3	10.5	380	0.98	0.0426	.8769	96.2	.178	2	.08	5.8	.0127	29.8
AA	10.5	360	1.00	0.0418	.8986	95.3	.140	105	.38	8.3	.0115	27.5
B-1	10.5	360	0.70	0.0448	.8832	96.4	.113	4	.05	5.7	.0133	29.7
B-2	10.5	370	0.72	0.0435	.8768	95.1	.115	9	.02	5.5	.0138	31.7
B-3	10.5	380	0.76	0.0412	.8697	92.7	.144	1	.02	7.8	.0140	33.9
C-1	10.5	360	0.49	0.0426	.8742	95.8	.112	1	.04	6.4	.0129	30.3
C-2	10.5	370	0.50	0.0418	.8693	95.5	.067	1	.04	7.1	.0138	33.0
C-3	10.5	380	0.50	0.0418	.8587	96.2	.068	0	.01	9.4	.0155	37.1
D-1	6.0	360	0.98	0.0426	.9018	95.7	.134	231	.02	5.2	.0098	23.0
D-2	6.0	370	1.01	0.0414	.9027	96.4	.173	188	.02	5.7	.0098	23.7
D-3	6.0	380	1.01	0.0414	.8989	95.4	.112	84	.02	6.3	.0096	23.2
E-1	6.0	360	0.74	0.0424	.9026	95.6	.220	158	.02	5.3	.0091	21.5
E-2	6.0	370	0.73	0.0429	.8972	94.1	.137	55	.02	6.0	.0073	17.0
E-3	6.0	380	0.73	0.0429	.8909	93.5	.080	18	.02	7.8	.0100	23.3
F-1	6.0	360	0.58	0.0360	.8959	86.9	.128	29	.01	5.2	.0084	23.3
F-2	6.0	370	0.51	0.0410	.8916	94.6	.075	20	.03	7.0	.0125	30.5
F-3	6.0	380	0.51	0.0410	.8853	93.6	.081	15	.07	8.4	.0128	31.2

CATALYST: Cyanamid HDS-20

TABLE 2:

HYDROTREATING SCREENING TESTS SUMMARY
STEARNS CATALYTIC/SRC Project 4046
DSS/CANMET File No.: 06SQ.23440-4-9318

RUN NO.	H/T CONDITION		FEED		SP GR	H/T LIQUID PRODUCT		GAS		H ₂ CONSUMPTION	
	P MPa	T °C	LHSV	H ₂ gmol/g		S Wt %	BASIC N WPPM	TW Wt %	YIELD Wt %	gmol/g	N %
A-1	10.5	360	0.92	0.0454	.9028	.376	320	.07	2.5	.0278	61.2
A-2	10.5	370	0.83	0.0504	.8952	.042	130	.08	4.7	.0148	29.4
A-3	10.5	380	1.00	0.0418	.8933	.052	94	.14	5.1	.0123	29.4
AA	10.5	360	0.96	0.0435	.9020	.131	335		4.4	.0109	25.1
B-1	10.5	360	0.84	0.0373	.8980	.106	60	.10	3.5	.0048	12.9
B-2	10.5	370	0.72	0.0435	.8916	.087	21	.06	5.0	.0119	27.3
B-3	10.5	380	0.73	0.0429	.8874	.070	39	.12	5.4	.0119	27.7
C-1	10.5	360	0.55	0.0380	.8867	.083	52	.21	5.0	.0160	42.1
C-2	10.5	370	0.49	0.0426	.8798	.063	21	.23	5.3	.0173	40.6
C-3	10.5	380	0.51	0.0410	.8846	.076	42	.21	5.4	.0167	40.8
D-1	6.0	360	0.90	0.0464	.9063	.139	494	.28	4.8	.0093	20.0
D-2	6.0	370	0.94	0.0445	.9032	.085	376	.12	5.7	.0099	22.2
D-3	6.0	380	0.96	0.0435	.9015	.085	277	.10	5.8	.0100	23.0
E-1	6.0	360	0.77	0.0407	.9042	.176	373	.10	5.3	.0094	23.1
E-2	6.0	370	0.74	0.0424	.8983	.097	183	.16	9.7	.0112	26.4
E-3	6.0	380	0.75	0.0418	.8946	.086	133	.25	7.9	.0106	25.4
F-1	6.0	360	0.53	0.0394	.9013	.256	73	.27	4.8	.0118	29.9
F-2	6.0	370	0.51	0.0410	.8959	.157	157	.46	6.5	.0112	27.3
F-3	6.0	380	0.50	0.0418	.8902	.056	78	.13	7.6	.0111	26.6

CATALYST: Cyanamid HDN-30

TABLE 3:

HYDROTREATING SCREENING TESTS SUMMARY
 STEARNS CATALYTIC/SRC Project 4046
 DSS/CANMET File No.: 06SQ.23440-4-9318

RUN NO.	H/T CONDITION		LHSV	FEED		SP GR	H/T LIQUID PRODUCT		TN	GAS YIELD	H ₂ CONSUMPTION	
	P MPa	T °C	-	H ₂ gmol/g	S Wt %	BASIC N WPPM	YIELD Wt %	Wt %		Wt %	gmol/g	N %
A-1	10.5	360	.98	0.0426	.310	315	96.1	.08	4.3	.0112	26.4	25.9
A-2	10.5	370	.99	0.0422	.194	199	95.7	.08	5.0	.0118	28.0	27.7
A-3	10.5	380	.98	0.0426	.076	129	96.2	.04	6.3	.0127	29.8	29.2
AA	10.5	360	.99	0.0422	.131	214	96.1	.04	5.5	.0098	23.2	23.0
B-1	10.5	360	.74	0.0424	.194	178	95.7	.06	4.4	.0123	29.0	28.6
B-2	10.5	370	.80	0.0392	.174	107	93.7	.06	4.3	.0121	30.9	32.9
B-3	10.5	380	.76	0.0412	.034	72	96.1	.08	6.1	.0141	34.2	34.7
C-1	10.5	360	.51	0.0410	.130	53	96.8	.09	4.2	.0144	35.1	35.8
C-2	10.5	370	.50	0.0418	.108	43	96.6	.11	5.7	.0156	37.3	37.3
C-3	10.5	380	.51	0.0410	.047	41	95.6	.04	6.0	.0159	38.8	39.5
D-1	6.0	360	1.01	0.0414	.351	652	97.6	.05	5.4	.0073	17.6	17.8
D-2	6.0	370	1.01	0.0414	.230	514	96.3	.08	6.3	.0079	19.1	19.3
D-3	6.0	380	1.01	0.0414	.156	317	95.4	.05	6.5	.0081	19.6	19.8
E-1	6.0	360	.76	0.0412	.278	471	95.2	.05	5.0	.0082	19.9	20.2
E-2	6.0	370	.75	0.0418	.171	299	95.6	.07	6.0	.0089	21.3	21.3
E-3	6.0	380	.79	0.0397	.097	173	95.0	.07	6.8	.0090	22.7	23.9
F-1	6.0	360	.56	0.0373	.254	255	97.2	.03	4.9	.0082	22.0	24.6
F-2	6.0	370	.52	0.0402	.158	132	95.8	.06	7.4	.0090	22.4	23.3
F-3	6.0	380	.53	0.0394	.032	64	94.0	.04	9.3	.0093	23.6	25.0

CATALYST: Haldor-Topsoe TK-581

TABLE 4:

 HYDROTREATING SCREENING TESTS SUMMARY
 STEARNS CATALYTIC/SRC Project 4046
 DSS/CANMET File No.: 06SQ.23440-4-9318

RUN NO.	H/T CONDITION		FEED		SP GR	H/T LIQUID PRODUCT		GAS YIELD	H ₂ CONSUMPTION	
	P MPa	T °C	LHSV	H ₂ g/mol/g		S Wt %	BASIC N WPPM		gmol/g	N %
A-1	10.5	360	0.97	0.0431	.9025	.293	200	4.4	.0115	26.7
A-2	10.5	370	0.96	0.0435	.8972	.204	80	4.9	.0124	28.5
A-3	10.5	380	1.01	0.0414	.8914	.118	46	5.3	.0127	30.7
AA	10.5	360	1.00	0.0418	.9045	.254	301	4.7	.0105	25.1
B-1	10.5	360	0.76	0.0412	.8699	.189	79	5.1	.0099	24.0
B-2	10.5	370	0.71	0.0441	.8899	.083	48	5.9	.0142	32.2
B-3	10.5	380	0.78	0.0402	.8825	.092	10	5.7	.0140	34.8
C-1	10.5	360	0.55	0.0380	.8887	.145	20	7.7	.0145	38.2
C-2	10.5	370	0.50	0.0418	.8806	.073	20	5.6	.0158	37.8
C-3	10.5	380	0.50	0.0418	.8715	.064	3			
D-1	6.0	360	1.03	0.0406	.9730	.296	556	4.6	.0102	25.1
D-2	6.0	370	1.02	0.0410	.9084	.301	565	5.2	.0104	25.4
D-3	6.0	380	1.01	0.0414	.9051	.208	375	5.8	.0111	26.8
E-1	6.0	360	0.79	0.0397	.9067	.359	412	3.9	.0110	27.7
E-2	6.0	370	0.75	0.0418	.9045	.298	306	5.6	.0116	27.8
E-3	6.0	380	0.74	0.0424	.8996	.201	169	7.3	.0122	28.8
F-1	6.0	360	0.55	0.0380	.9044	.385	248	5.1	.0091	24.0
F-2	6.0	370	0.52	0.0402	.8994	.227	135	7.6	.0099	24.6
F-3	6.0	380	0.51	0.0410	.8927	.127	68	8.3	.0104	25.4

CATALYST: Ketjen KP-840

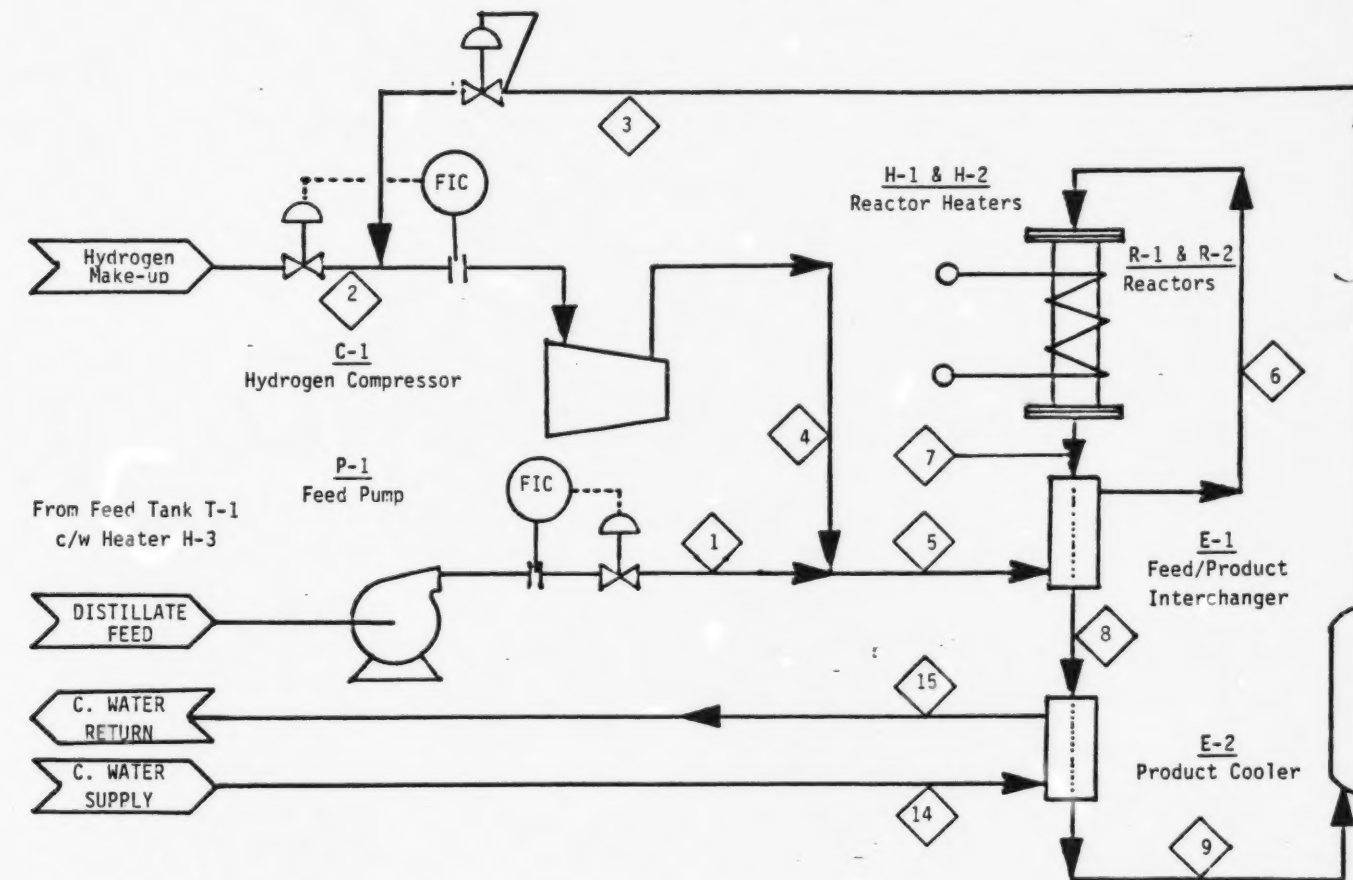
TABLE 5:

 HYDROTREATING SCREENING TESTS SUMMARY
 STEARNS CATALYTIC/SRC Project 4046
 DSS/CANMET File No.: 06SQ.23440-4-9318

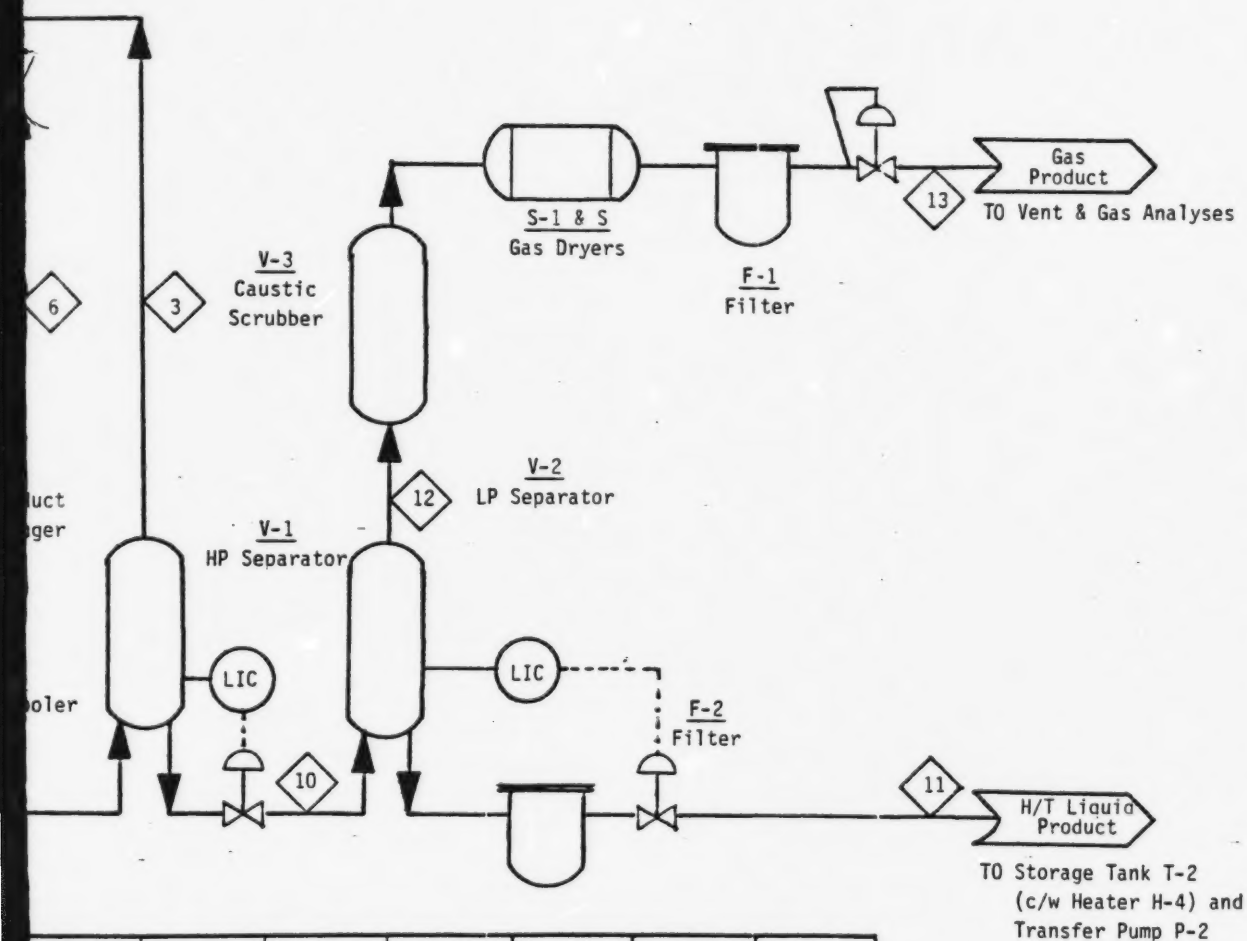
RUN NO.	H/T CONDITION		LHSV	FEED	SP GR	H/T LIQUID PRODUCT		GAS YIELD	H ₂ CONSUMPTION	
	P MPa	T °C	-	H ₂ gmol/g	-	YIELD Wt %	S Wt %	TN Wt %	gmol/g	N %
A-1	10.5	360	1.02	0.0410	.8976	84.1	.273	.35	.0220	53.7
A-2	10.5	370	0.98	0.0426	.8911	96.4	.178	.30	.0202	47.4
A-3	10.5	380	1.08	0.0387	.8870	84.9	.090	.20	.0187	48.3
AA	10.5	360	0.97	0.0431	.8997	95.9	.210	.44	.0183	42.5
B-1	10.5	360	0.73	0.0429	.8908	96.0	.181	.12	.0225	52.4
B-2	10.5	370	0.71	0.0441	.8830	96.2	.115	.13	.0234	53.0
B-3	10.5	380	0.72	0.0435	.8710	94.8	.177	.40	.0242	55.6
C-1	10.5	360	0.52	0.0402	.8849	95.8	.168	.40	.0256	63.7
C-2	10.5	370	0.51	0.0410	.8973	95.8	.162	.30	.0260	63.5
C-3	10.5	380	0.52	0.0402	.8730	97.0	.133	.04	.0277	68.9
D-1	6.0	360	1.01	0.0414	.9050	95.8	.267	.20	.0101	24.4
D-2	6.0	370	1.00	0.0418	.9048	95.7	.202	.20	.0106	25.4
D-3	6.0	380	0.99	0.0422	.9006	96.5	.128	.16	.0110	26.1
E-1	6.0	360	0.82	0.0382	.9045	97.6	.246	.30	.0081	21.2
E-2	6.0	370	0.75	0.0418	.9000	97.8	.123	.20	.0127	30.4
E-3	6.0	380	0.80	0.0392	.8974	85.4	.204	.09	.0121	30.9
F-1	6.0	360	0.53	0.0394	.9005	96.2	.195	.43	.0107	27.1
F-2	6.0	370	0.50	0.0418	.8933	94.9	.118	.36	.0118	28.2
F-3	6.00	380	0.53	0.0394	.8879	95.2	.106	.43	.0115	29.2

C. PROCESS FLOW DIAGRAM (PFD) - CANADIAN HYDROTREATING TEST CENTRE

DRAWING NO. 080-B3000



 STREAM NO. STREAM TITLE		1	2	3	4	5	6	7	8
		DISTILLATE FEED	HYDROGEN FEED			REACTOR FEED		HP REACTOR PRO	
			MAKE-UP	RECYCLED	TOTAL	COLD	HEATED	HOT	INTER.
PRESSURE	MPa	6.0-10.5	0.21	6.0-10.5	6.0-10.5	6.0-10.5		6.0-10.5	
TEMPERATURE	°C	65	20	80	59	64.8	190	390	220
FLOW - LIQUID	Kg/hr	2.884	-	-	-	2.884		2.769	
SULPHUR	Wt %	3.5	-	-	-	3.5		0.2	
- H ₂ S GAS	S1/hr	-	-	-	-	-		66.12	
- OTHER GASES	S1/hr	-	-	12.82	12.82	12.82		32.88	
- HYDROGEN	S1/hr	-	872	1874	2746	2746		1874	
NOTES		Based on LHSV = 1.0; R-1 Vol. = 3.0 l; H ₂ Feed Set at 0.0425 gmol/g-Liq. Feed; Dist							



D. PIPING & INSTRUMENTATION DIAGRAM (P & ID) - CANADIAN
HYDROTREATING TEST CENTRE

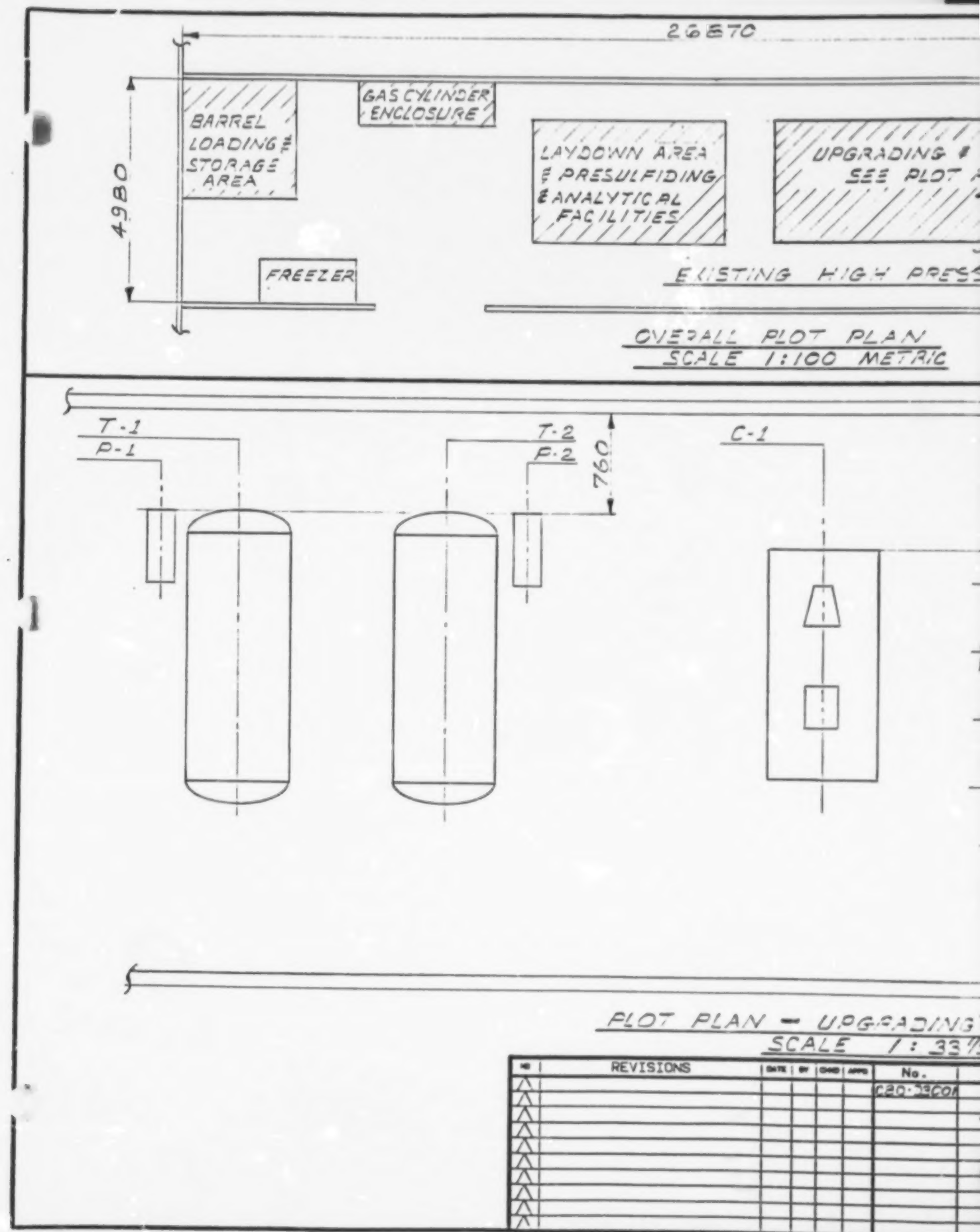
DRAWING NO. 080-D3001

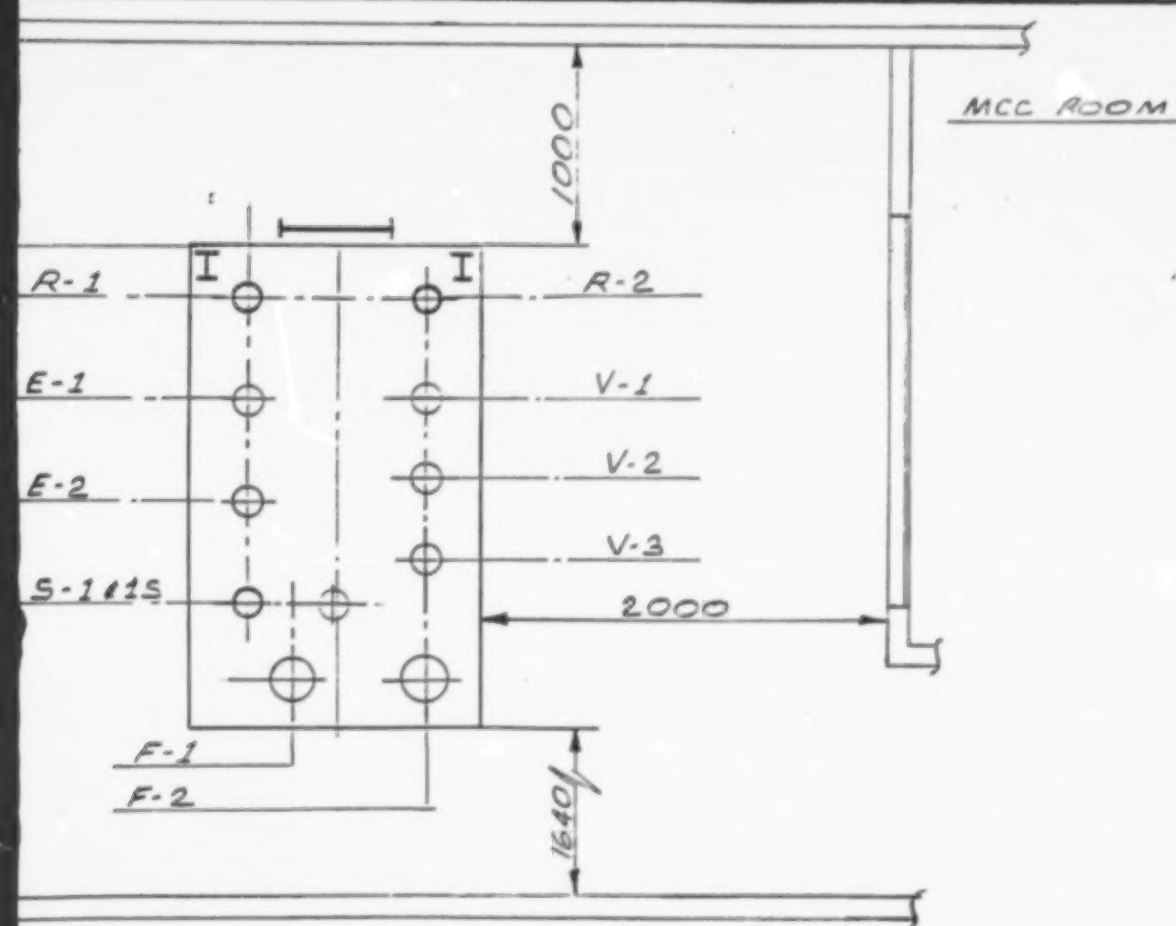
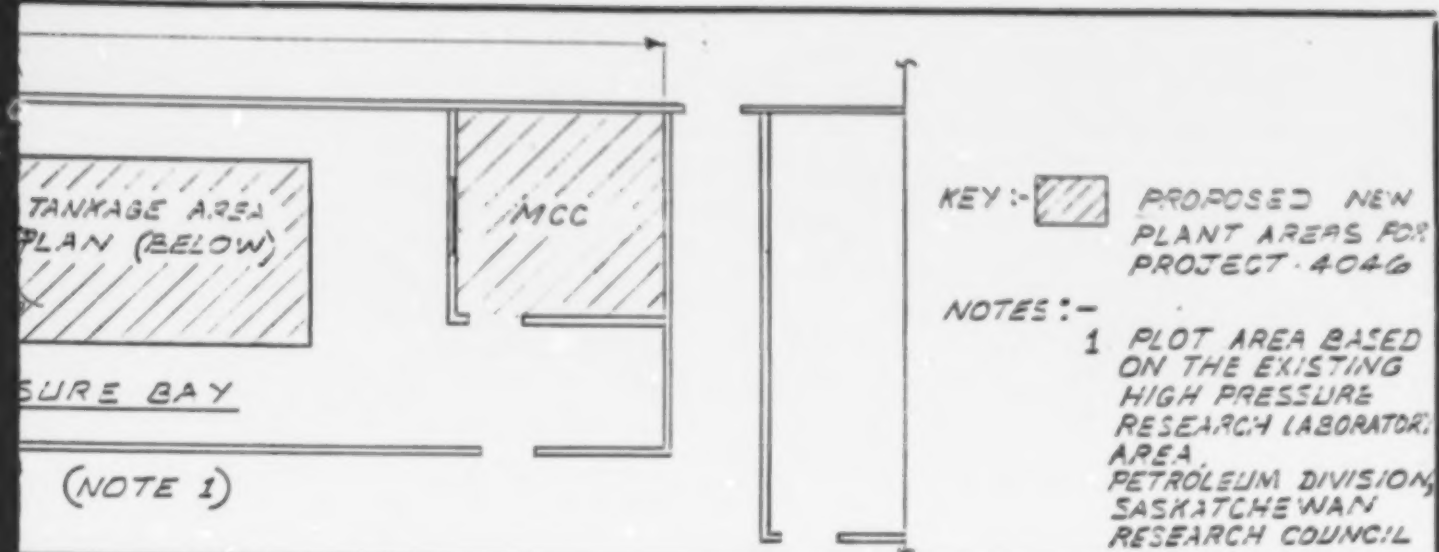
8		9	10	11	12	13	14 15
REACTOR PRODUCT		LP REACTOR	H/T LIQUID	GAS PRODUCT		C. WATER	
INTER.	COOLED	PRODUCT	PRODUCT	UNTREATED	TREATED	SUPPLY/RET	
6.0-10.5		0.21	0.11	0.21	0.11	0.5/0.45	
220	80	80	80	80	70	18/68	
2.769		2.769	2.769	-	-	6.03	
0.2		0.2	0.2	-	-	-	
66.12		66.12	-	66.12	-	-	
32.88		20.06	-	20.06	20.06	-	
1874		-	-	-	-	-	

Feed; Distillate Sp. Gr. = 0.9614

CANADIAN HYDROTREATING TEST CENTRE
PROCESS FLOW DIAGRAM
STEARNS CATALYTIC PROJECT 4046
DSS/ERL FILE: 06SQ.23440-4-9318
UPGRADING OF SYNTHETIC CRUDE DISTILLATES
WITH COMMERCIAL HYDROTREATING CATALYSTS
DWG. NO. 080-B3000
Stearns Catalytic

E. PLOT PLAN - CANADIAN HYDROTREATING TEST CENTRE
DRAWING NO. 140-B2001





TANKAGE AREA
1/3 METRIC

REFERENCE DRAWINGS	ENG. RECORD	DRAWING STATUS	ENERGY RESEARCH LABORATORY CANMET OTTAWA	DSS/EMR FILE No. 06SQ.23440-4-931E
P110	DRAWN P.H.	DRAWN		
	CHECKED	PRELIMINARY		
	REVIEWED	FOR APPROVAL		
	EXTRACTED	APPROVED	CANADIAN HYDROTREATING CENTRE	CLIENT NO.
	SELECTED		UPGRADING OF SYNTHETIC CRUDE DISTILLATES WITH COMMERCIAL HYDROTREATING CATALYSTS	PROJECT NO. 4046
	PROPOSED		PLOT PLAN	S.E.L. FILE NO. 140-B2001
	PROJECTED		Stearns Catalytic	SCALE AS NOTED
	DESIGNED			REVISION Δ
	PREPARED			DATE FILED

F. EQUIPMENT LIST - CANADIAN HYDROTREATING TEST CENTRE



EQUIPMENT LIST

C-1 HYDROGEN RECYCLE COMPRESSOR

Service: Compress and deliver make-up and recycle hydrogen

Gas Flow: Make-up Hydrogen 1,750 sl/hr
Recycle Hydrogen 3,750 sl/hr
Total 5,500 sl/hr
Design 6,000 sl/hr

Composition: 99.5% H₂, 0.2% CH₄, 0.2% N₂ and 0.1% C₂H₆

Average M.Wt: 2.265

Suction Condition: 210 kPa, 59°C

Discharge Head: 15.5 MPa; 18.6 MPa (Rated)

E-1 PRODUCTS/FEED INTERCHANGER

Service: Exchanging heat between high temperature hydrotreated reactor products stream and cool hydrogen-liquid feed.

Type of Exchanger: Double-pipe, concentrically jacketted.

Thermal Duty: 0.5 kW, nominal; 1.0 KW design

Inner Pipe: 1" NPS - Reactor Products

Outer Pipe: 2" NPS - Hydrogen/Liquid Feed Mixture

Temperatures - Inner: 390°C in, 200°C out
- Outer: 59°C in, 190°C out

Overall Heat Transfer Coefficient: 0.06 kW/°C m², estimated

E-2 PRODUCT COOLER

Service: Final cooling of hydrotreated reactor product.

Type of Exchanger: Double-pipe, concentrically jacketted.

Thermal Duty: 0.5 kW, nominal; 1.0 kW, design

EQUIPMENT LIST (cont'd)

E-2 PRODUCT COOLER (cont'd)

Inner Pipe: 1" NPS - Reactor Products

Outer Pipe: 2" NPS - Cooling Water

Temperatures - Inner: 220°C in, 80°C (max.) out

- Outer: 18°C in, 68°C (max.) out

Overall Heat Transfer Coefficient: 0.15 kW/°C m², estimated

F-1 ENTRAINMENT FILTER

Service: Filtering exhaust hydrocarbon gas mixture from dryer.

Type: Cartridge, in-line filter

Flow: 20 sl/hr., nominal; 30 sl/hr design

Gas Composition: 70% C₂, 20% C₃, 10% C₄ and C₅

Entrainment: Solid particulates from dryer bed

Particle Size: Maximum pass-through size 5 microns

Pressure: 210 kPa operating; 420 kPa, design

Temperature: 80°C, operating; 120°C design

F-2 LP PRODUCT FILTER

Service: Filtering low pressure hydrotreated liquid product.

Type: Cartridge, in-line

Flow: 2.77 kg/hr, nominal, 4.2 kg/hr, design

Liquid Property: 0.90 Sp. Gr., 50-500 m.Pa.Sec viscosity

Particle size: maximum pass-through size 1.0 microns

EQUIPMENT LIST (cont'd)

F-2 LP PRODUCT FILTER (cont'd)

Maximum Pressure Drop: 35 kPa

Pressure: 210 kPa, operating; 420 kPa, design

Temperature: 80°C, operating; 120°C, design

H-1 HYDROTREATING REACTOR HEATERS

(A, B, C, D and E)

Service: Reactor preheater and auxiliary reaction heating.

Type: Spiral Wound Induction Heater, insulated.

Reactor Dimension: 1.315" OD x 4'L per section per Heater

Material: stainless steel

Thickness: 0.179" (0.455 cm)

Insulation: Calcium Silicate, 2" thickness, min.

Temperature (skin): 495°C

Electrical Power Output: 0.5 kW (each Heater)

Supply: VAC, 220/110 V/60 HZ/3 phases

H-2 PRE-HYDROTREATING REACTOR HEATERS

(A, B and C)

(Identical to H-1)

H-3 FEED IMMERSION HEATER

Service: Heating of hydrotreater feedstock in Feed Tank (T-1)

Type: Chromolox electrical, single element, low intensity with sheath

Feedstock Property: 0.98 Sp. Gr., 2500 m.Pa.S at 20°C, 100 m.Pa.S at 150°C; 5% sulphur content

Thermal Duty: 25 kW, nominal

Tank Dimension: 2'6" ID x 6'S/S

EQUIPMENT LIST (cont'd)

H-4 PRODUCT IMMERSION HEATER

Service: Heating of hydrotreated liquid product in Product Tank T-2

H/T Liquid Product Property: 0.9 Sp. Gr., 2,500 m.Pa.S at 20°C, 50 m.Pa.S at 150°C; 0.5% maximum sulphur content

Type, Duty and Tank Dimension: Identical to H-3

P-1 FEED PUMP

Service: Feedstock pumping from Feed Tank (T-1) to process system

Fluid and Property: Hydrotreating feed stocks (Hydrocarbon liquid)

- Flowrate 1.45 - 5.76 kg/hr.
- Sp. Gr. 0.87 - 0.98
- Viscosity 500 - 2,500 m.Pa.S at 20°C
- Vapour pressure 0.5 kPa maximum
- Sulphur 0.9 - 5 wt%
- Pour Point 0°C - 70°C

Suction Condition: From Feed Tank (T-1) with Heater (H-3) on

- Temperature 100°C nominal (pumping temperature)
- Static P 0.6 m (2') minimum
- NPSH_a 9.7 m (32')

Discharge Pressure: 6.0 MPa, minimum; 15 MPa, maximum; 18.0 MPa, design

Type of Pump: Plunger or Double-diaphragm complete with electrical or pneumatic stroke adjustment

EQUIPMENT LIST (cont'd)

P-2 HYDROTREATED PRODUCT TRANSFER PUMP

Service: Transfer of hydrotreated product to drum, barrel or downstream processing units.

Fluid and Property: Hydrocarbon liquid

- Flowrate 15 kg/min. max.
- Sp. Gr. 0.8 - 0.9
- Viscosity 500 - 2,500 m.Pa.S at 20°C, 50-250 m.Pa.S at 150°C
- Vapour pressure 0.5 kPa max.
- Sulphur 0.5 wt% max.
- Pour point 0°C - 70°C

Suction Condition: From Storage Tank (T-2) with Heater (H-4) on:

- Pumping Temperature 100°C max.
- Static P 0.5 m (1.6')
- NPSH_a 9.5 m (31')

Discharge Pressure: 300 kPa (for FCC feed) to 150 kPa (drum or barrel filling)

Type of Pump: Centrifugal or Diaphragm

P-3 BARREL TRANSFER PUMP (not identified in P & ID)

Service: Transfer of barrel/drum content to Feed Tank (T-1)

Fluid and Property: Hydrotreater feed (hydrocarbon liquid)

- Flowrate 17 kg/min., nominal
- Sp. Gr. 0.87 - 0.98
- Viscosity 500-2,500 m.Pa.S
- Vapour pressure 0 kPa
- Sulphur 0.9 - 5 wt%

Suction and Discharge Conditions:

- Pumping temperature 20°C (room temperature)
- NPSH_a 7.5 m (25')
- Discharge P 150 kPa

Type of Pump: Pneumatic and Portable complete with air-driven self-lubrication and strainer

Air Pressure: Supply at 700 kPa (plant air) maximum

EQUIPMENT LIST (cont'd)

R-1 HYDROTREATING REACTOR

Service: Catalytic hydrotreating reaction of hydrogen and hydrocarbon liquid mixture

Feedrate and Composition:

- Hydrogen Gas - 2,46 sl/hr.
- H/C Liquid - 2.884 kg/hr., Sp. Gr. = 0.95
- Sulphur in Liquid - 1.0 to 5.0 wt%
- Other Gases - 12.8 2 sl/hr. (N_2 and CH_4)

Temperature: 390°C, nominal; 450°C, design

Pressure: 15 MPa, nominal; 18 MPa, design

Type: Downward, Trickle, Catalytic Reactor

Construction: Sectional 1" NPS x 4'H per section (maximum 5), sections to be flanged ends

R-2 PRE-HYDROTREATING REACTOR

Identical to R-1, except that maximum number of sections to be 3.

S-1 & S GAS DRYER

Service: Drying of scrubbed low pressure H/C gases

Flow: 20.1 sl/hr., saturated with water; design to 30 sl/hr.

Temperature: 70°C, nominal; 85°C, design

Pressure: 0.21 MPa, nominal; 0.31 MPa, design

Pressure Drop: 35 kPa maximum

T-1 FEED TANK

Service: Receive and store hydrotreating feed liquid

Feed Property: Hydrocarbon Liquid

- Sp. Gr. 0.98
- Viscosity 2,500 m.Pa.S at 20°C
- Sulphur 5% max.

EQUIPMENT LIST (cont'd)

T-1 FEED TANK (cont'd)

Temperature: 150°C maximum, 180°C design

Pressure: Atmospheric

Volume and Dimension: 2'6" ID x 6'S/S, 830 l

Nozzles and Instruments:

- Breather
- PSV
- TI and LI
- 1" Feed Nozzle, 2" steam-out and 1" pump-out nozzles plus 2" drain valve

T-2 PRODUCT STORAGE TANK

Service: Receive and store hydrotreated liquid

Product Property: Hydrocarbon liquid; Sp. Gr. 0.90, viscosity 2,500 m.Pa.S at 20°C, sulphur 0.5%

Other Design: Identical to T-1

V-1 HP SEPARATOR

Service: Separation of high pressure (HP) gas and liquid mixture

Flow and Composition:

- 1,874 sl/hr Hydrogen
- 32,88 sl/hr C₁, C₂ and N₂
- 66.12 sl/hr (Hydrogen) sulphides
- 3.1 sl/hr Hydrocarbon liquid

Temperature: 80°C, nominal; 220°C, design

Pressure: 15 MPa, maximum; 18 MPa, design

Volume and Dimension: 4" NPS x 20" H (T/T), 4 litre, pipe-capped ends

EQUIPMENT LIST (cont'd)

V-1 HP SEPARATOR (cont'd)

Nozzles and Instruments:

- 1" inlet, pipe 12" above bottom
- 3/4" liquid outlet
- 1" gas outlet
- 1" PSV connection
- 1" LIC connection

V-2 LP SEPARATOR

Service: Separation of low pressure (LP) gas and liquid mixture

Flow and Composition:

- 66.12 sl/hr sulphides (Gas)
- 20.1 sl/hr (C₃, C₄ and C₅)
- 3.1 sl/hr Hydrocarbon Liquid

Temperature: 80°C, nominal; 220°C, design

Pressure: 0.21 MPa, nominal; 0.31 MPa, design

Volume, Dimension, Nozzles and Instruments: Identical to V-1

V-3 CAUSTIC SCRUBBER

Service: Removal by chemical absorption of sulphides

Solution: Caustic solution, Sp. Gr. 0.95; 60% filled

Gas Flow: 66.12 sl/hr sulphides gas and 20.1 sl/hr light H/C gas

Temperature: 80°C (gas inlet); 120°C, design

Pressure: 0.21 MPa, nominal; 0.31 MPa, design

Volume and Dimension: 4" NPS x 10" T/T, 2 litres

1

G. PROJECT STAFF LIST

- o STEARNS CATALYTIC LTD., CALGARY, ALBERTA
 - o PETROLEUM DIVISION, SASKATCHEWAN
RESEARCH COUNCIL, REGINA, SASKATCHEWAN
- 2

PROJECT STAFF LIST

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R.D. (Roy) Hutchinson	-	Sr. Piping Designer
D.A. (Don) Roberts	-	Sr. Instrumentation Technologist
G.S. (Paul) Sarpal	-	Sr. Mechanical Engineer

Petroleum Division, SRC, Regina, Saskatchewan

Subcontract Manager: D.W. (Doug) Soveran

C. Jackson	-	Research Technologist
R. Ranganathan	-	Project Advisor
E. Ogu	-	Research Engineer
N. Snatilla	-	Research Technologist

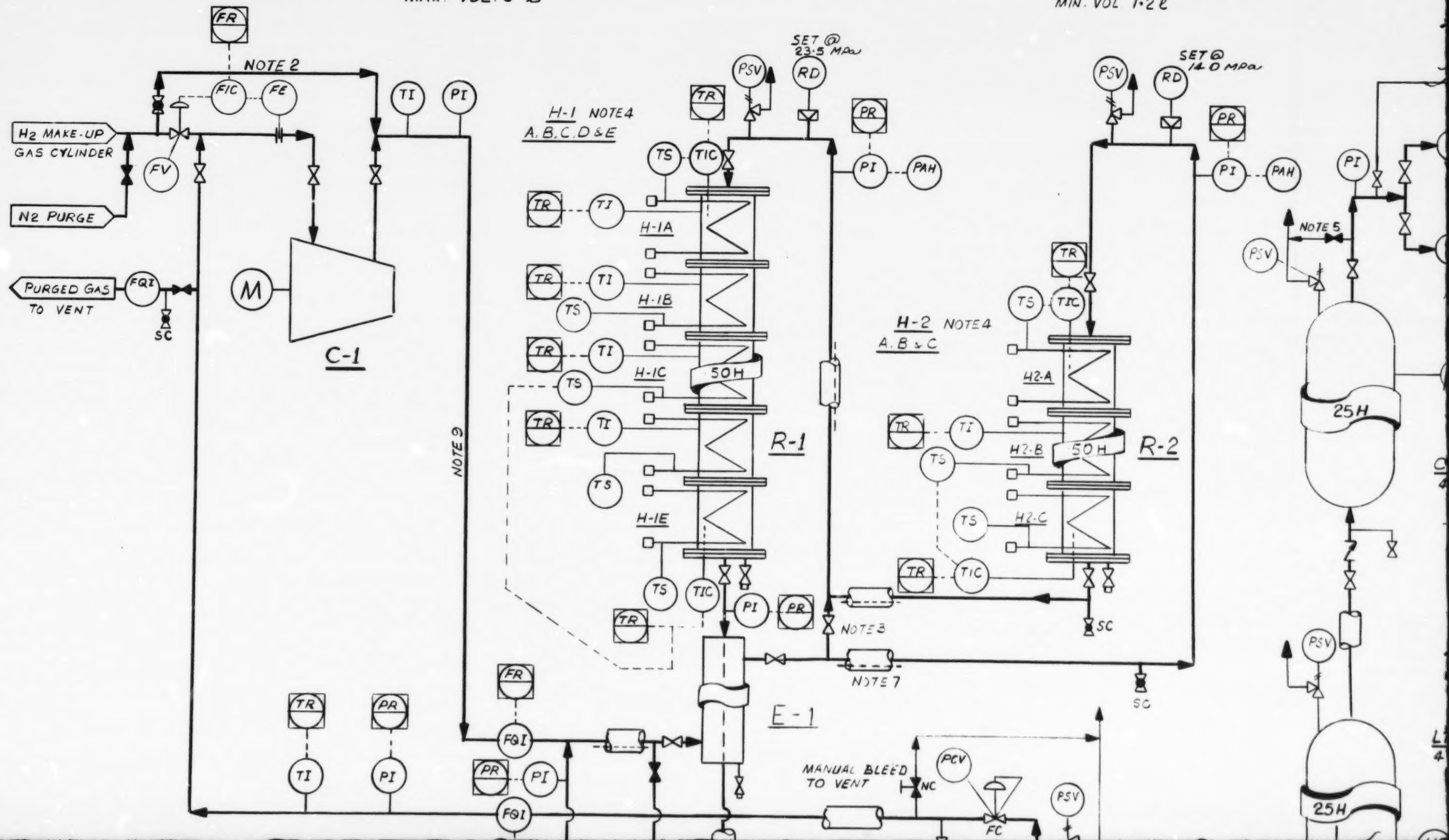
C-1
HYDROGEN RECYCLE COMPRESSOR
 6000 SL HR, 3.53 SCFM, 18.6 MPa

R-1
HYDROTREATING REACTOR
 1" I.D. x 4'-0" HIGH SECTION
 MAX. 5 SECTIONS
 MAX. VOL. 3 L

H-1 A B C D E
HT REACTOR HEATERS
 0.5 KW @

R-2
PRETREATING REACTOR
 1" I.D. x 4'-0" HIGH/SECTION
 MIN. 2 SECTIONS
 MIN. VOL. 1.2 L

H-2 A B
PRE-H T REACTO
 0.5 KW @

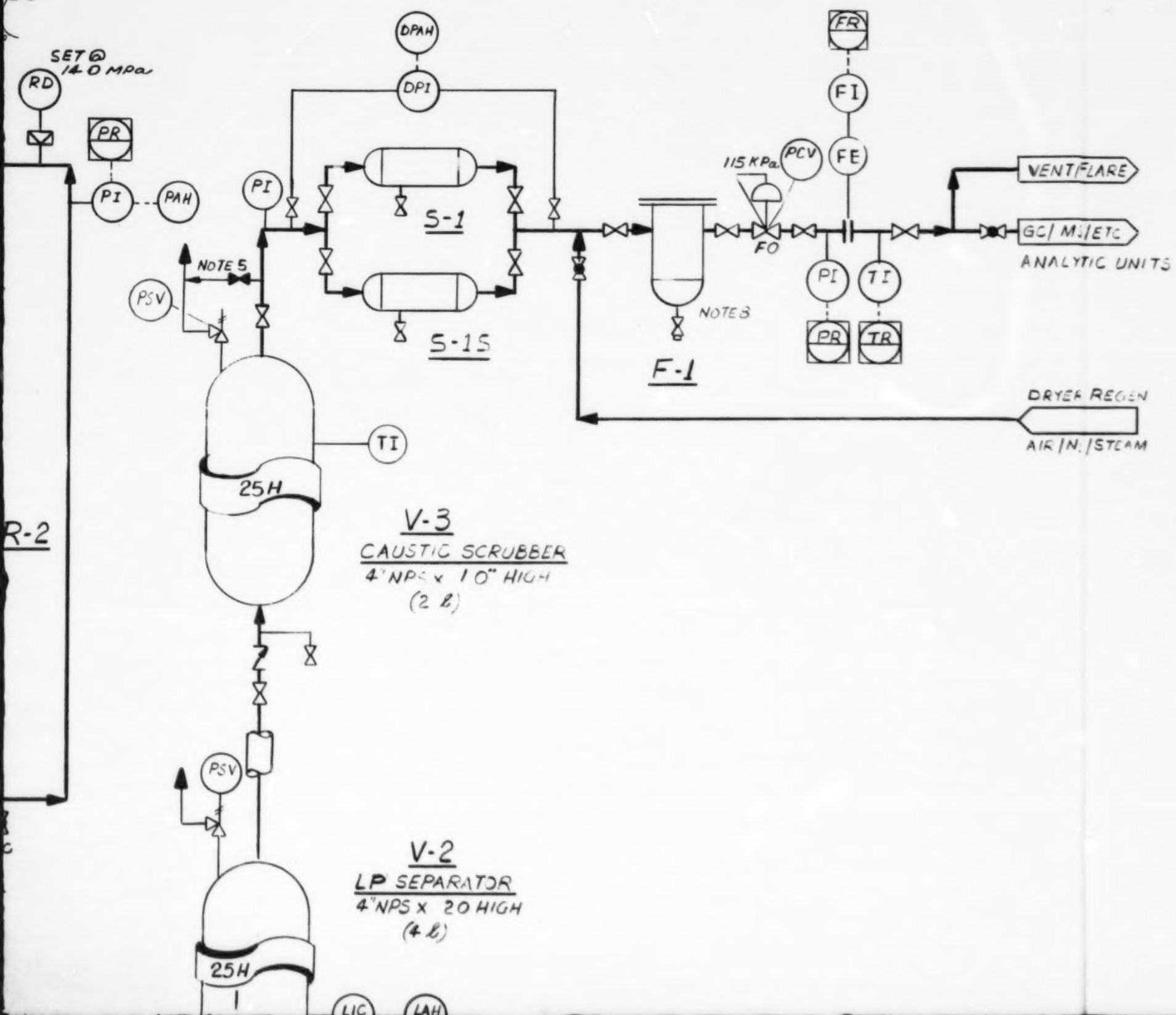


REACTOR
SECTION
IONS
22

H-2 A B & C
PRE-H T REACTOR HEATERS
0.5 KW @

S-1 & 1S
GAS DRYERS

F-1
ENTRAINMENT
FILTER

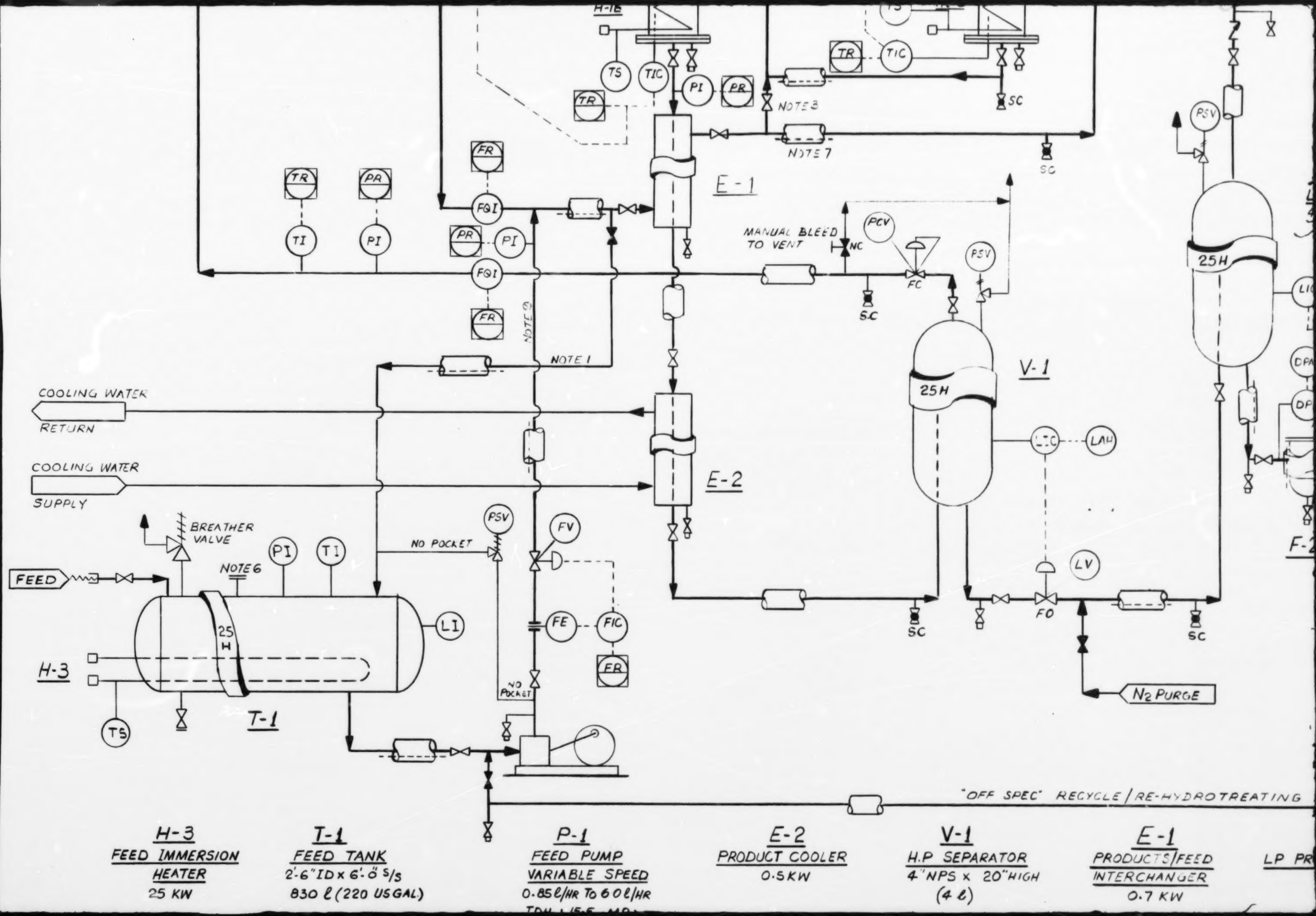


NOTES:

1. FEED PUMP START-UP RECIRCULATION LOOP.
2. START-UP HYDROGEN FEED BY-PASS
3. DIRECT HYDROTREATING TO R-1 FOR FEEDS THAT DO NOT REQUIRE PRETREATING FOR REMOVING CATALYST POISONS
4. H-1 AND H-2 ARE INDUCTION HEATERS, C/W TRANSFORMER
5. DRYER REGENERATION GAS DISCHARGE
6. STEAM-OUT CONNECTION
7. LINES UPSTREAM OF R-1 TRACED AND INSULATED
8. CRUDS OR ENTRAINMENT FILTERS TO PROTECT CONTROL VALVES AND FLOW ELEMENT
9. FOR EASE OF LINE SUPPORT, MINIMUM 3/4" NPS LINE SIZE TO BE USED FOR ALL LINES, EXCEPT FOR LOCAL SHORT RUN LINES

INSTRUMENT SYMBOLS

- | | |
|-------|----------------------------------|
| F | = FLOW |
| L | = LEVEL |
| P | = PRESSURE |
| DP | = DIFFERENTIAL PRESSURE |
| T | = TEMPERATURE |
| E | = ELEMENT |
| I | = INDICATOR |
| IC | = INDICATING CONTROLLER |
| AH | = HIGH ALARM/ANNUNCIATOR |
| (F)QI | = ON-LINE FLOWMETERING INDICATOR |
| R | = RECORDER (TO COMPUTER) |
| RD | = RUPTURE DISC |
| S | = SWITCH |
| TS | = TEMPERATURE SWITCH |
| CV | = CONTROL VALVE |



INSTRUMENT SYMBOLS

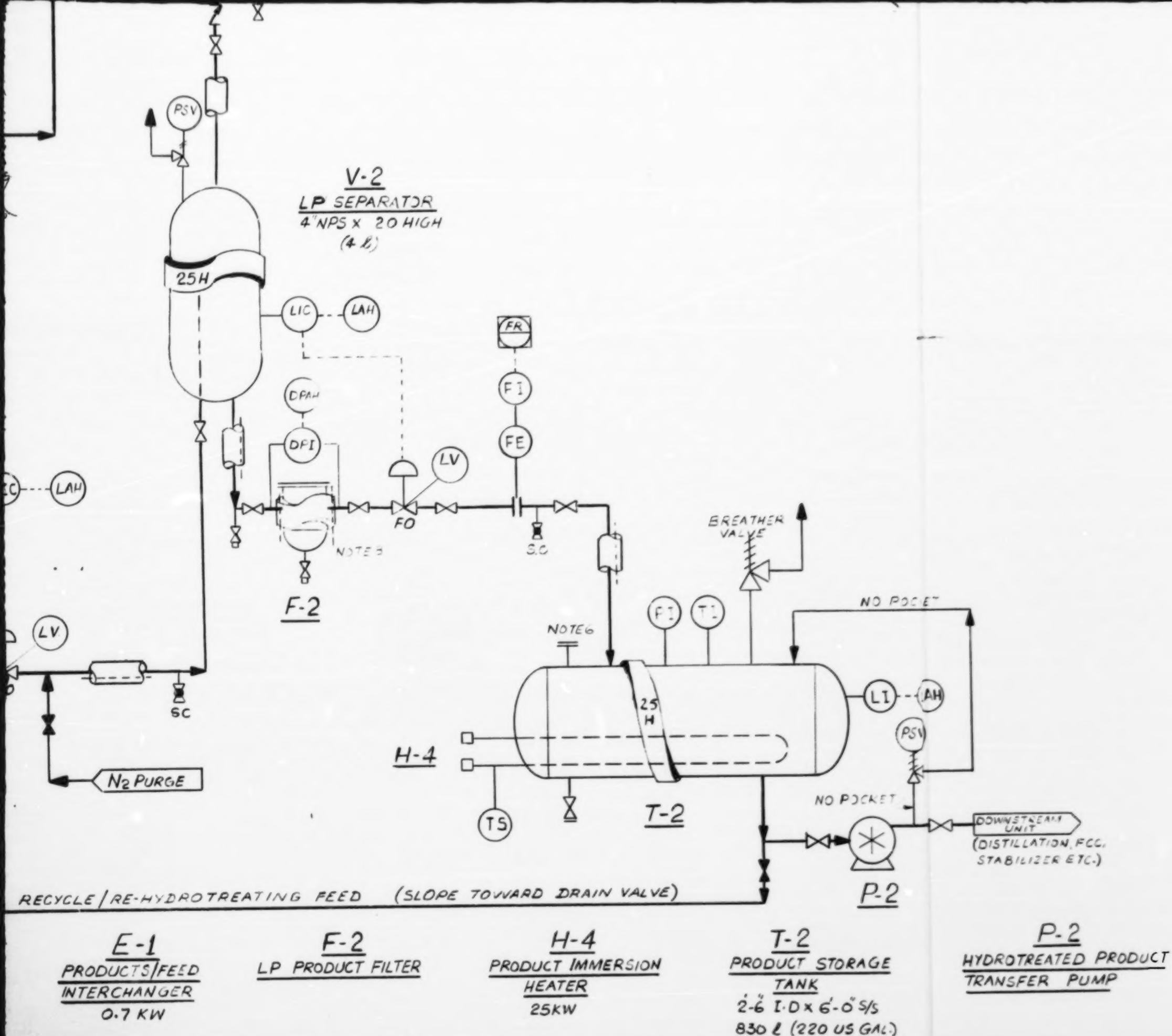
F	= FLOW
L	= LEVEL
P	= PRESSURE
DP	= DIFFERENTIAL PRESSURE
T	= TEMPERATURE
E	= ELEMENT
I	= INDICATOR
IC	= INDICATING CONTROLLER
AH	= HIGH ALARM/ANNUNCIATOR
(F)QI	= ON-LINE FLOWMETERING INDICATOR
R	= RECORDER (TO COMPUTER)
RD	= RUPTURE DISC
S	= SWITCH
TS	= TEMPERATURE SWITCH
CV	= CONTROL VALVE
SV	= SAFETY VALVE
V	= VALVE

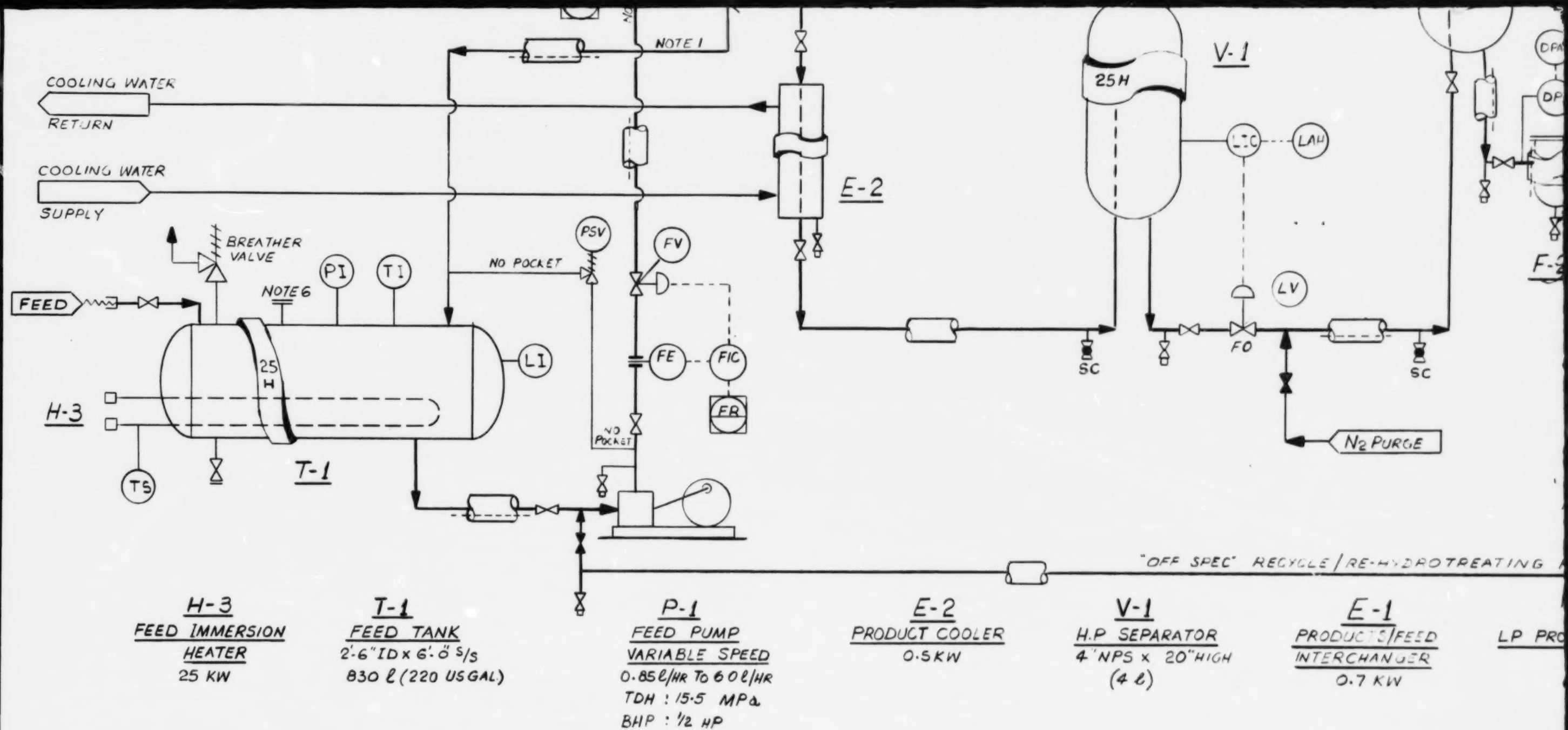
---  INPUT TO COMPUTER

NOTATIONS

	= NORMALLY CLOSED VALVE
 SC	= SAMPLE CONNECTION
25H/50H	= INSULATION, 25 MM/50 MM THICKNESS
NPS	= NOMINAL PIPE SIZE

PRESULFIDING FACILITY AND CONNECTION NOT SHOWN





NO.	REVISIONS	DATE	BY	CH'D.	APP'D.	ENGINEERING REVIEW		AUTHORIZED P
A	ISSUED FOR REVIEW & COMMENTS	86-8-8	MA	J.W.	J.W.	DISCIPLINE	APP'D. BY/DATE	
O	ISSUED AS A PROJECT DOCUMENT	86 11 19	RH			CONTROL SYSTEMS		
						ELECTRICAL		PROCESS ENGINEER
						EQUIPMENT		PROJECT ENGINEER
						PIPING ENG.		
						PIPING DES.		
						PROCESS ENG.		

ANY PROCESS CONDITIONS OF S
TURES, PRESSURES, FLOW QU
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GRAM ARE CALCULATED AND A
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THEY DO NOT REPRESENT EXACT
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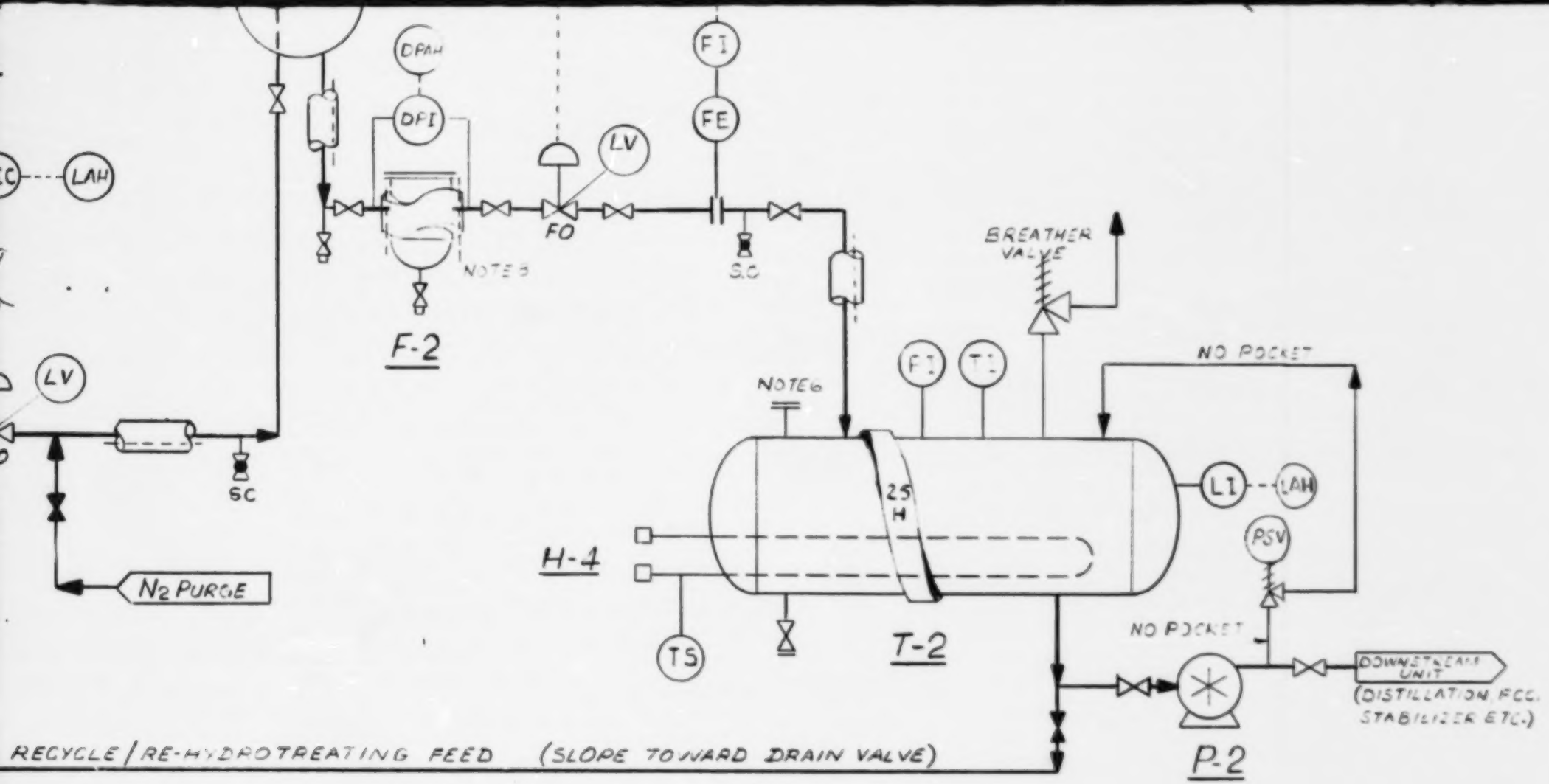
GUARANTEES ARE CONTAINED C
PROPOSAL DOCUMENTS

-----  INPUT TO COMPUTER

NOTATIONS

-  = NORMALLY CLOSED VALVE
-  SC = SAMPLE CONNECTION
- 25H/50H = INSULATION, 25 MM/50 MM THICKNESS
- NPS = NOMINAL PIPE SIZE

PRESULFIDING FACILITY AND CONNECTION NOT SHOWN



E-1
PRODUCTS/FEED
INTERCHANGER
0.7 KW

F-2
LP. PRODUCT FILTER

H-4
PRODUCT IMMERSION
HEATER
25KW

T-2
PRODUCT STORAGE
TANK
2'-6" I.D x 6'-0" S/S
830 L (220 US GAL.)

P-2
HYDROTREATED PRODUCT
TRANSFER PUMP

DRAWING REVIEW		AUTHORIZED FOR RELEASE		CUSTOMER APPROVAL		DRAWING STATUS			ENERGY RESEARCH LABORATORY CANMET OTTAWA		THIS DOCUMENT IS THE EXCLUSIVE PROPERTY OF STEARNS CATALYTIC LTD. IT HAS NOT BEEN PUBLISHED AND IS CONFIDENTIAL IN CONSIDERATION OF THE LOAN HEREOF. RECIPIENT PROMISES NOT TO COPY, REPRODUCE, EXHIBIT, LOAN OR OTHERWISE FURNISH OR DISPOSE OF NOR TO USE THIS DOCUMENT FOR ANY PURPOSE OTHER THAN THAT FOR WHICH IT IS SPECIFICALLY FURNISHED WITHOUT THE WRITTEN PERMISSION OF STEARNS CATALYTIC LTD. NOT TO USE THE SAME IN ANY WAY DETRIMENTAL TO STEARNS CATALYTIC LTD. CLIENT NO. DSS PROJ/ FILE 06SQ.23440-4-9318 PROJECT NO. 4046 S.C.L. DWG. NO. 080-D3001 DRAWN CHECKED REVISION
APP'D. BY/DATE		APP'D. BY	DATE			ISSUE NO.	APP'D.	DATE	PRELIMINARY PIPING & INSTRUMENTATION DIAGRAM CANADIAN HYDROTREATING TEST CENTRE		
						DEVELOPMENT					
						PRELIMINARY ENGINEERING					
						FINAL ENGINEERING					
		PROCESS ENGINEER				APPROVED FOR CONSTRUCTION			UPGRADING OF SYNTHETIC CRUDE DISTILLATES WITH COMMERCIAL HYDROTREATING CATALYSTS		
		PROJECT ENGINEER				REV 					
		ANY PROCESS CONDITIONS OF SERVICE, SUCH AS TEMPERATURES, PRESSURES, FLOW QUANTITIES, STREAM COMPOSITIONS, AND EQUIPMENT DUTIES, SHOWN ON THIS FLOW DIAGRAM ARE CALCULATED AND ARE FOR DESIGN PURPOSES ONLY. WHILE THEY MAY BE USEFUL AS GUIDES IN OPERATION, THEY DO NOT REPRESENT EXACT OR GUARANTEED OPERATING CONDITIONS.				NOT APPROVED FOR CONSTRUCTION UNLESS SIGNED AND DATED. DESTROY ALL PRINTS BEARING EARLIER DATE AND/OR REVISION NO.					
		GUARANTEES ARE CONTAINED ONLY IN THE CONTRACT (OR PROPOSAL) DOCUMENTS			BY	DATE					